The Preparatory Manual of Explosives: Radical, Extreme, Experimental Explosives Chemistry

Vol.1

A comprehensive look at a variety of radical explosives that are currently not in use or are being investigated.

A book by Jared Ledgard

Note: None of the explosives listed in this book have been thoroughly investigated and are considered "non existent". Only trained professionals should attempt to duplicate any information contained in this book.

This book is designed to express ideas and theories for possible reactions and products.

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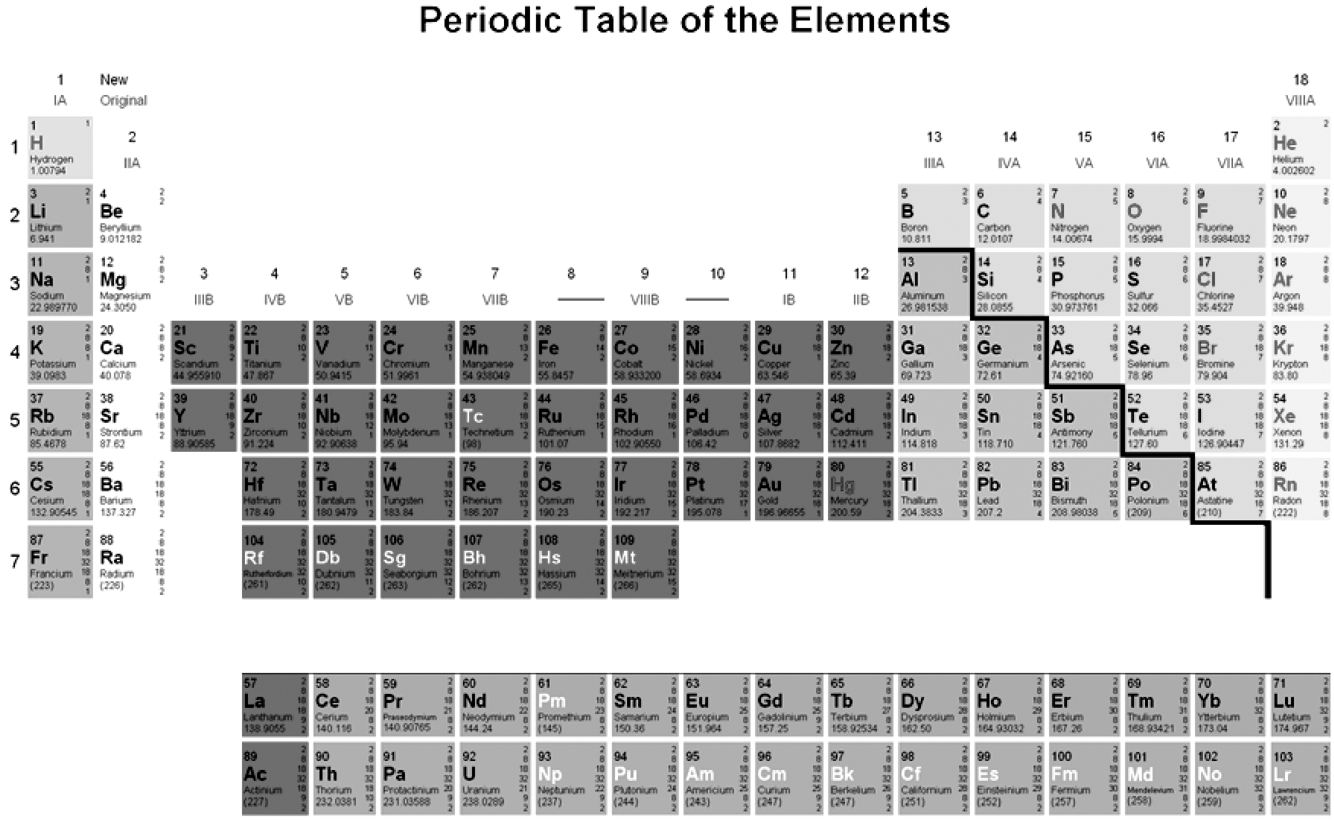
Section 1: Introduction

In this book you will experience a branch of chemistry that is very exclusive and interesting, but is not a part of normal chemical research. This book will discuss the theology and ideology of experimental chemistry in a way never before seen in any other book to this date circa 2010. Radical experimental chemical synthesis is very rare, and does not fit into the norm of ordinary pharmaceutical chemistry where by chemists conduct research on new drugs using a variety of tests, chemical functional groups, and other reactions that are very common and based on proven published works found in the American chemical Society, the Asian and European chemical abstracts, and the chemical abstract service (CAS). However, in this book, the experimental chemistry is based on theoretical ideas of chemical reactions that may or may not take place. It should be known that the experiments in this book have not been thoroughly tested or investigated, and the experiments in this book are based on ideas and theological formulas.

First of all as with many of my books, we should start off with a brief description of chemistry and laboratory techniques.



Notice on the above image of the periodic table how the elements are represented by single letters or double letters. Don't get confused about the elements that are represented by single letters and those represented by double letters. There is nothing special about the elements represented by single letters or double letters and it's solely arbitrary. Notice how each element is arranged in columns and rows. This does have significant meaning, as each column or row of elements have similarities to them, meaning the elements in each column or row for example, have similar physical properties. These similar physical properties can be vast in definition, so they will not be discussed here.
In many cases, the letter representing the corresponding element matches the first letter or so of the name of the element. For example, the element oxygen is represented by the letter "O". Also, notice how letters "Al" represent the element Aluminum; however, this representation is not true for all elements. For example, the element Tungsten is represented by the letter "W", and the element Tin is represented by the letters "Sn".

Now that you have an idea about elements, and the four major categories of chemistry and a few of their compounds, its time to discuss the foundation to chemistry, and that foundation is the nature of "chemical reactions". What is a chemical reaction, and why does a chemical reaction take place? Chemical reactions take place because of many different reasons; however, a few of those reasons will be discussed here. As you have seen, the four main categories of chemistry are inorganic chemistry,

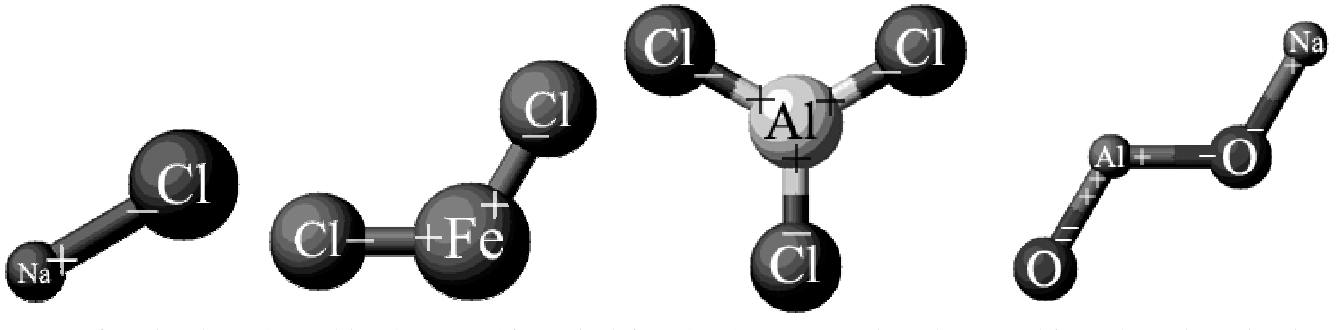
organic chemistry, biochemistry, and nuclear chemistry; however, we will only discuss the nature of inorganic and organic reactions that take place.
In the nature of chemical reactions we will only deal with inorganic and organic reactions and we will not discuss the reactions of bioorganic compounds or nuclear reactions as these are to complex and complicated for this book. Inorganic compounds and organic compounds under go chemical reactions based on two types of “bonding”. This bonding utilizes two major types of chemical bonding, 1) ionic bonding, and 2) covalent bonding. Remember these two types of bonding, as they are very very important in understanding the nature of chemical reactions.

Ionic bonding

Ionic bonding is the simplest form of chemical interaction. In an ionic compound, the atoms within the molecule are held together by positive and negative charges. These charges are what form ionic compounds. If we look at sodium chloride for example, the ionic bond is between the positive sodium atom, and the negative chlorine atom. So why is the sodium positive and the chlorine negative? This is answered by discussing electro negativities of the various atoms, meaning each atom or element, possesses its own unique electro negativity. The element sodium has a really low electro negativity, whereby the element chlorine has a rather high electro negativity; therefore, the element sodium possesses positive energy, and the chlorine atom possesses negative energy. In essence, because the chlorine possesses negative energies, and the sodium possesses positive energies, when the two atoms, or elements come in contact with one another, they chemical react, and form a ionic bond, i.e., the chlorine swipes one of the sodium’s electrons to fulfill its electrical demands. When an atom looses an electron from its nucleus, it becomes positively charged. At the same time, on the other end, when an atom gains an electron from a weaker atom, it in-itself becomes negatively charged. Lets look at the following table:

Atom (element)	Electro negativity
Lithium	1.00
Sodium	1.00
Magnesium	1.20
Calcium	1.0
Titanium	1.30
Iron	1.60
Copper	1.80
Aluminum	1.50
Lead	1.60
Nitrogen	3.10
Oxygen	3.50
Sulfur	2.40
Fluorine	4.10
Chlorine	2.8
Iodine	2.2

You will notice by looking at the table that the element sodium possesses an electro negativity of 1.0, and the element chlorine posses an electro negativity of 2.8. Because the difference between the electro negativities is greater then 1.5, the compound sodium chloride is ionic. Lets look at a few examples of ionic compounds.

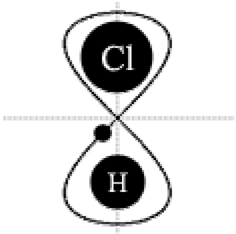


Far left molecule: sodium chloride; second from the left molecule: Ferrous chloride; second from the right molecule: Aluminum chloride; and far right molecule: sodium aluminate. Notice how the number of positives equals the number of negatives.

As previously mentioned, when sodium and chlorine react with each other, the chlorine swipes one of the sodium's electrons, thus making sodium positive and chlorine negative. The reason this happens is because the element sodium has a weak electrical charge. Because sodium has such a weak charge, and the chlorine as a more powerful electrical charge, the sodium must give up an electron to the chlorine. If this sounds confusing, think about two magnets, and how two magnets stick together when exposed to opposite poles. When two magnets are placed side-by-side with identical poles, they repel each other. Ionic compounds work in the same manner. Obviously identical charges would repel each other, therefore, when ionic compounds are formed, their positive and negative charges attract each other, but in order for this to happen, each atom must either gain or give-up electrons. As briefly mentioned before, when an atom gives-up electrons, it becomes positive, and when an atom gains electrons, it becomes negative. Therefore, in every single case, when one atom gives-up an electron, another atom gains it simultaneously. As noticed in the above illustration atoms can give-up and gain more then one electron. Notice the second from the right molecule, i.e., aluminum chloride. Aluminum is capable of giving up three electrons, and hence becoming a positively charged atom with a positive charge of 3. At the same time, you need three negative charges to balance things out, and hence, the aluminum bonds with three chlorine atoms, each chlorine atom has a negative charge. In this case, each chlorine atom only swipes one of the aluminums three available electrons. So why does the chlorine atom only swipe one electron, and why does aluminum give-up three electrons. This is a good question and will be discussed later.

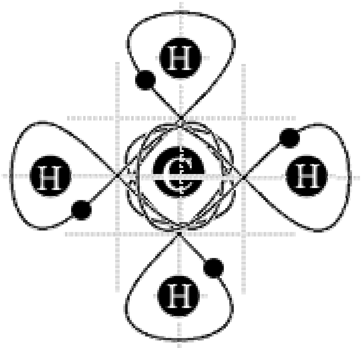
Covalent bonding

As you have learned, ionic compounds form their bonds by utilizing differences in electro negativities. Covalent compounds on the other hand, form their bonds by sharing electrons rather then taking them, or giving them up. So what exactly does this mean? Well, in sharing, the electrons orbit around each atom of the molecule that makes up the bond. If this sounds confusing, lets study the following illustration.



Covalent bonding structure of hydrogen chloride. As with covalent bonding the electrons form figure eight patterns around each other. This is how the atoms in the molecule share their electrons.

As seen in the above illustration, the electrons in the molecule orbit around both atoms using a figure eight type orbital pattern. This is the foundation to covalent bonding. Now lets look at a more difficult covalently bonded system in the following illustration.

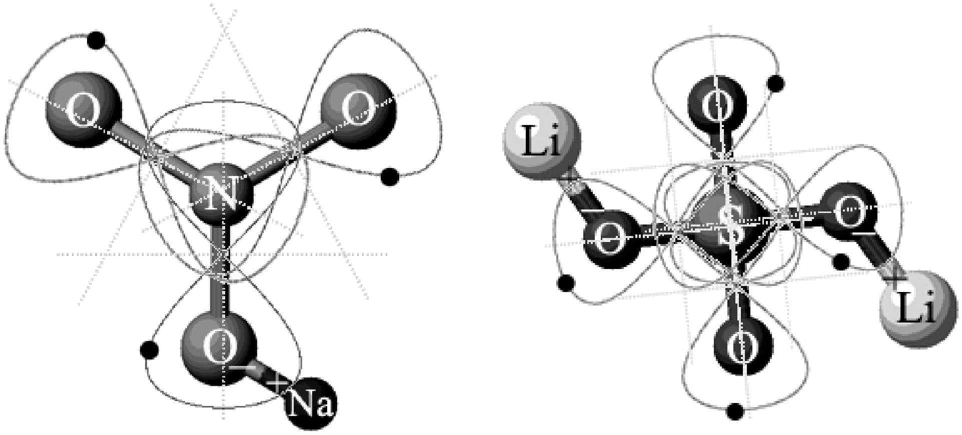


In this illustration we see the molecule methane, where four hydrogen atoms are covalently bonded to a central carbon atom whereby the electrons use figure eight orbital patterns. This is another great example of the sharing of electrons that takes place in covalent bonding.

In covalent bonding, the difference between electro negativities of the atoms usually does not exceed 1.50. What this means is that when atoms with differences in electro negativities are less 1.50, the bonding is said to be covalent. For example, when we see the above structure of hydrogen chloride, the hydrogen has an electro negativity of 2.2, and the chlorine atom has an electro negativity of 2.8, making the difference between the two electro negativities 0.60. Because this difference of 0.60 is less then 1.50, the molecule is covalent. The bonding in the majority of organic compounds is covalent in nature.

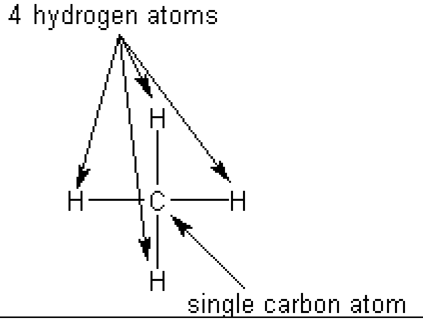
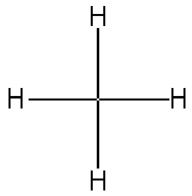
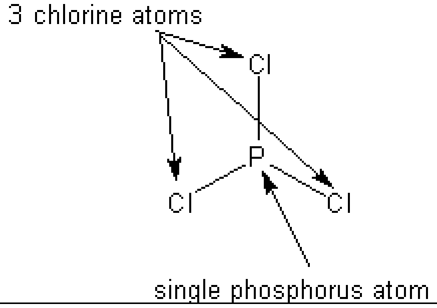
Dual bonding

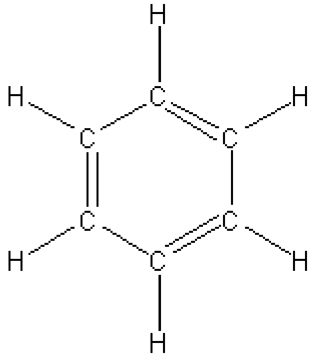
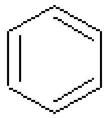
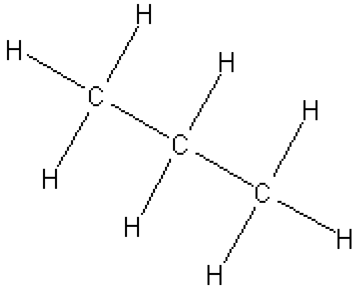
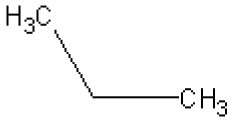
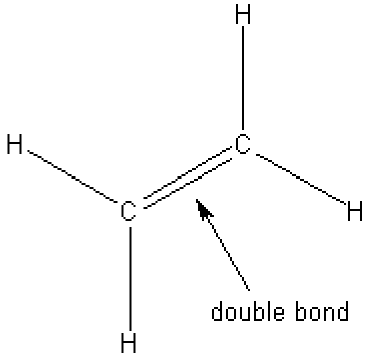
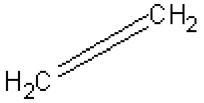
In some cases, molecules will be composed of both ionic, and covalent bonding. Not to confuse you, but remember that if the difference between electro negativities between two atoms is less then 1.50, the bonding is covalent, and if the difference is greater then 1.50, the bonding is predominantly covalent. Therefore, molecules will often have dual bonding, composed of both ionic and covalent bonds. For a better example, lets look at the following illustrations:



In the left illustration the molecule sodium nitrate contains both an ionic bond between the oxygen atom and the sodium atom, and three covalent bonds between the central nitrogen atom and the three adjacent oxygen atoms. In the right illustration we see the molecule lithium sulfate, with two ionic bonds, each from the lithium atom to a single oxygen, and we see four covalent bonds between the central sulfur atom, and the four adjacent oxygen atoms.

Before we discuss the oxidation states of the various elements, we need to take a quick lesson into chemical structures and formulas. In chemistry, we use chemical structures and formulas to explain what is taking place during a chemical reaction, and to explain to the reader what chemical is being talked about. When writing structures and formulas, we use letters to represent the atoms involved, and we use lines to represent the bonding between the various atoms involved. Therefore, lets get familiarized with the chemical structures and their formulas by looking at the following table:

Expanded chemical structure	**Condensed chemical structure**	**Condensed structural formula**
 4 hydrogen atoms H H C H H single carbon atom		CH_4
The expanded structure of methane. Notice how four hydrogen atoms are covalently bonded to a central carbon atom.	***The condensed chemical structure of methane.***	***Condensed structural formula***
 3 chlorine atoms Cl P Cl Cl single phosphorus atom	PCl_3	PCl_3

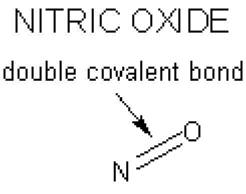
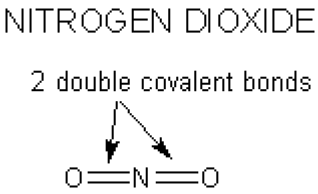
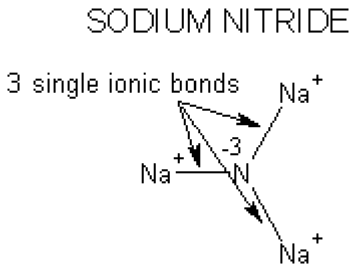
The expanded structure of phosphorus trichloride.	***The condensed chemical structure of phosphorus trichloride.***	***Condensed structural formula***
 6 carbon ring, containing 6 carbon atoms 6 hydrogen atoms, each one bonded to a single carbon atom		C_6H_6
The expanded structure of benzene. Notice how every other carbon is double bonded to the next one.	***Condensed chemical structure of benzene***	***Condensed structural formula***
 3 carbon chain, 8 hydrogen atoms		C_3H_8
The expanded structure of propane. Each hydrogen bonded to a single carbon atom.	***Condensed chemical structure of propane***	***Condensed structural formula***
 2 carbon chain; 6 hydrogen atoms		C_2H_4
The expanded structure of Ethene. Notice the double bond between the two carbon atoms. Each hydrogen is bonded to a single carbon atom.	***Condensed chemical structure of Ethene***	***Condensed structural formula***

Now that you have a rough-cut idea about atoms, chemical bonding, and structure, its time to understand why chemical reactions take place in the first place. Each atom has what are called oxidation states. Oxidation states are closely related to the number of bonds an individual atom can form. These oxidation states dictate the atoms within a molecule, and defines the what, when, how, and why they chemically react with and why the reactions take place between atoms and molecules in the first place. As briefly mentioned in section 1b under ionic bonding, aluminum likes to give up three electrons. So why is this the case? The answer is oxidation states. Aluminum has an oxidation state of +3, meaning when it reacts with other elements or when it reacts with molecules, it always involves three electrons in the reaction. In other words, three electrons will always be given up if the aluminum is involved in a chemical reaction involving ionic compounds, or it will always share three electrons if involved in a chemical reaction involving covalent compounds. The oxidation state of aluminum will always be +3 and nothing will change that. Therefore, lets get familiarized with the oxidation states of the most common elements. Note: Some elements have only one oxidation state, and some have more then one oxidation state.

Element	**Oxidation State(s)**	**Number of bonds formed**
Hydrogen (H)	+1, -1	1
Sodium (Na)	+1	1
Magnesium (Mg)	+2	2
Calcium (Ca)	+2	2
Iron (Fe)	+2, +3	2 and 3
Copper (Cu)	+1, +2	1 and 2
Aluminum (Al)	+3	3
Carbon (C)	+2, -4, +4	2, and 4
Nitrogen (N)	+1, -1, +2, -2, +3, -3, +4, +5	1, 2, 3, 4, and 5
Oxygen (O)	-2	2
Phosphorus (P)	+3, -3, +5	3 and 5
Sulfur (S)	-2, +4, +6	2, 4, and 6
Chlorine (Cl)	+1, -1, +5, +7	1, 5, and 7
Iodine (I)	+1, -1, +5, +7	1, 5, and 7

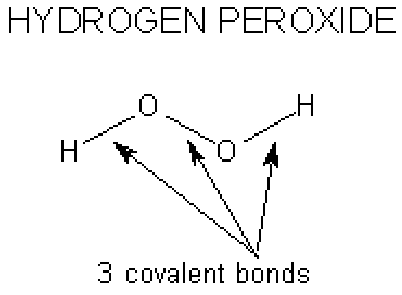
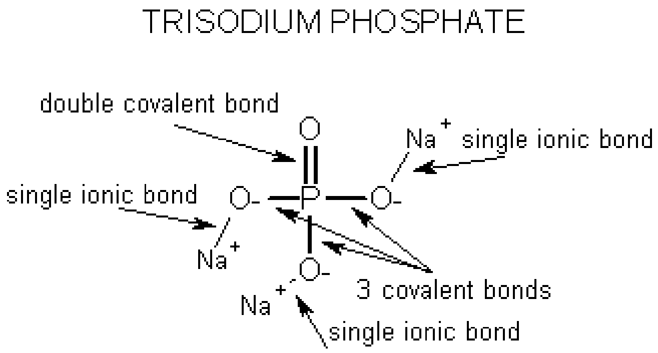
Now, understanding the oxidation states of the various elements can be confusing, but what it really comes down to is simple memorization. However, we could go into much greater detail to why the elements form their oxidations states, but it would require some pretty advanced information, which will not be discussed in this book. In the meantime however, lets focus on several elements, such as Nitrogen, Oxygen, and Chlorine. When you zoom in on these elements, you will notice the various oxidation states that they have, and the corresponding number of bonds they can form. Remember that the number of oxidation states in each element is closely related to the number of bonds the element can form; therefore, lets focus on the following tables for examples:

The Element Nitrogen

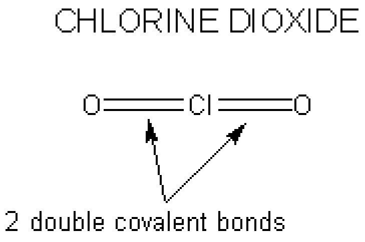
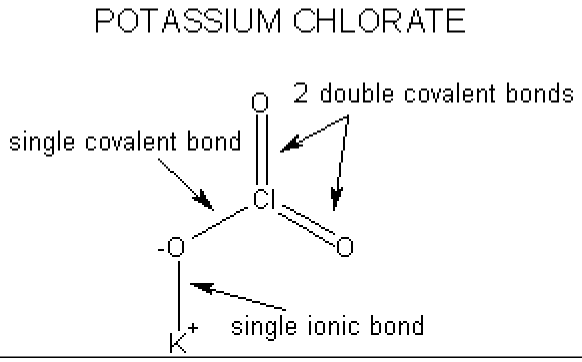
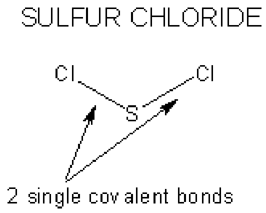
Structure example	**Structure example**	**Structure example**
		 SODIUM NITRIDE 3 single ionic bonds Na+ -3 Na+ N Na+
1. Nitrogen is in the +2 oxidation state 2. Oxygen is in the –2 oxidation state	***1. Nitrogen is in the +4 oxidation state 2. Oxygen is in the –2 oxidation state***	***1. Nitrogen is in the –3 oxidation state 2. Sodium is in +1 oxidation state***
Because the nitrogen is in the +2 oxidation state, and its electro negativity is only slightly smaller then the oxygen, it forms a double covalent bond with the oxygen, which is in the –2 oxidation state. In other words, nitrogen and oxygen both form 2 bonds. Note how the positive and negative oxidation charges are in relation to the number of total bonds formed, i.e., +2 and –2. Don't confuse these charges with ionic bonding	Because the nitrogen is in the +4 oxidation state, and its electro negativity is only slightly smaller then the oxygen, it forms two double covalent bonds with the oxygens, each of which have –2 oxidation state. In other words, the nitrogen forms 4 bonds, and the two oxygen atoms both form 2 bonds. Note how the positive and negative oxidation charges are in relation to the number of total bonds formed, i.e., +4 and –4. Don't confuse	In this case, the nitrogen is in the –3 oxidation state because its electro negativity is greater then 1.5 over the sodium. Therefore, its electrical charges are in exact relation to one another, i.e., +3 and –3. Each sodium atom is bonded to the negatively charged nitrogen atom using ionic bonding.

	these charges with ionic bonding.	

The element oxygen

Structure example	Structure example
	 TRISODIUM PHOSPHATE double covalent bond O Na+ single ionic bond single ionic bond O- P O- Na+ O- Na+ 3 covalent bonds single ionic bond
1. Oxygen is in the –2 oxidation state ***2. Hydrogen is in +1 oxidation state***	***1. Oxygen is in the –2 oxidation state*** ***2. Phosphorus is in the +5 oxidation state*** ***3. Sodium is in +1 oxidation state***
In this example, the oxygen has a –2 oxidation state, and the hydrogens have a +1 oxidation state each. Because the difference in electro negativities is less then 1.5, the bonds are covalent. In this example, the two oxygen atoms form 2 bonds each, and the hydrogen forms 1 bond each. Notice how the two oxygen atoms share a single covalent bond.	In this example, we have three different atoms bonded in the same molecule. We see a central phosphorus atom, which is in a +5 oxidation state. Because its electro negativity is less then 1.5 as compared to the oxygen atoms, it forms 5 bonds. To do so, it forms a double covalent bond with a single oxygen and three single covalent bonds between three other oxygen atoms. On the other end of these three oxygen atoms, we have three single ionic bonds formed to three sodium atoms as the result of the sodium atoms difference in electro negativity, being more then 1.5.

The element chlorine

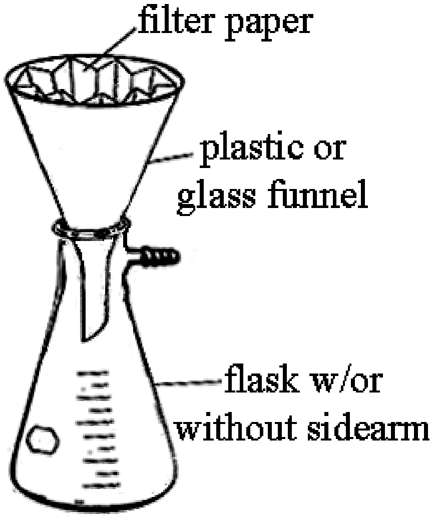
Structure example	Structure example	Structure example
	 POTASSIUM CHLORATE O 2 double covalent bonds single covalent bond Cl -O O K+ single ionic bond	
1. Chlorine is in +4 oxidation state ***2. Oxygen is in the –2 oxidation state***	***1. Chlorine is in the +5 oxidation state*** ***2. Oxygen is in the –2 oxidation state*** ***3. Potassium is in the +1 oxidation state***	***1. Chlorine is in the –1 oxidation state*** ***2. Sulfur is in the +2 oxidation state***
In this example, the chlorine is in the +4 oxidation state therefore it forms 4 bonds. In doing so, it forms 2 double covalent bonds with two oxygen atoms, both of which have a –2 oxidation state, and hence, forming 2 bonds each. Notice how the number of bonds balances out, i.e., 4 bonds to 4 bonds total.	In this example, the chlorine is in a +5 oxidation state, therefore, it forms 5 bonds. In doing so, it forms two double covalent bonds with two oxygen atoms, and a single covalent bond with a third oxygen atom. This third oxygen in turn, satisfies its 2 bond needs by forming an ionic bond between a potassium atom because its difference in electro negativity is greater then 1.5.	In this simple example, two chlorine atoms are forming 1 bond each, so in doing so, they are bonded to a single sulfur atom, which requires 2 bonds because of its +2 oxidation state.

Introduction to filtration

Filtration is a rather simple process and involves two different forms, gravity filtration and vacuum filtration (suction filtration). In either case, filtration is a necessary tool used in chemistry and many other applications to recover insoluble or precipitated matter or products. Filtration utilizes a filter media, or also called filter paper, where the insoluble or precipitated matter or products collects on. Filter paper is specially designed with micro small holes so that insoluble matter or products collects, but liquids and solutions are allowed to pass through. Note: Many people use filtration in their daily lives, such as when you brew up a cup of coffee, you place the coffee into a filter, and then hot water passes there through; in essence, the act of preparing a cup of coffee is an extraction process, and a filtration process in one.

1) Gravity filtration

Gravity filtration is the oldest form of filtration and requires a filter, which utilizes gravity to pull the liquid through the micro small holes of the filter paper. Gravity filtration is the most common form of filtration in general labs, but its use should be limited due to the severe slowness of the filtration, especially when trying to filter very fine precipitated matter. When filtering very fine precipitated matter, or also called particles, the gravity filtration process can take hours even days. To carryout a gravity filtration process, the liquid to be filtered is poured into the plastic or glass funnel, and then you have to wait for all the liquid to drain through the filter, leaving the insoluble material collected on the filter paper. Once the insoluble material has been collected, and the liquid has thoroughly passed through the filter, you can collect the insoluble mass from the filter paper. Note: the insoluble mass on the filter paper will often be moist with the liquid that you filtered, and this can be a pain to deal with, so you will need to wash the collected material, see section 2f to learn about this.



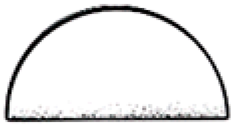
Classic setup for gravity filtration.

Instead of using gravity filtration to recover a precipitated product, it is sometimes used to purify liquids and mixtures by the use of a filter aid. A filter aid is a insoluble solid material that is placed inside the filter paper prior to use. To use, the desired mixture or solution to be purified is simply poured into the filter and allowed to drain and collect into the flask. The filter aid is designed to absorb impurities from the mixture or solution, or in most cases a reaction mixture. The filter aid ranges from granulated charcoal, activated carbon, silicon dioxide, silica gel, sand, zeolites, diatomaceous earth, or even aluminum oxide.

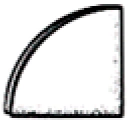
Fluting Filter Paper for use in gravity filtration

When carrying out gravity filtration, glass or plastic cones or funnels are often used. In this regards, filter paper, which is often sold in circular form must be fluted and formed to fit properly in the glass or plastic cone or funnel. Before you flute your filter paper, the size of the circular filter paper to be used should be at least 2 times the diameter of the top of the cone or funnel your using for the filtration. Note: In gravity filtrations, fluted filter paper is often superior to other forms of filtering paper because the “flutedness” of the filter paper allows for better airflow between the glass or plastic cone or funnel wall, and the fluted filter paper. To flute standard circular filter paper:

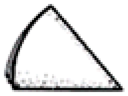
1. Take three circular sheets of filter paper, lay them on top of each other, and then fold them in half.



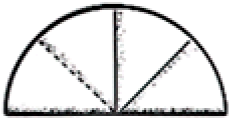
2. Next, take this half, and fold it into quarters, i.e, fold this portion into half.



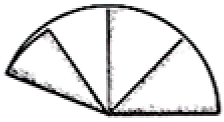
3. Fold this portion into eighths, i.e, fold this halved portion into half's.



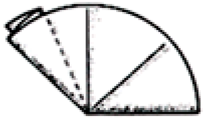
4. Open the halved filter paper up, to the original half-folded position.



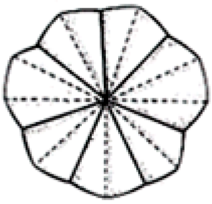
5. Take the opened filter paper, and fold to the eighth line.



6. Then fold to the quarter line.

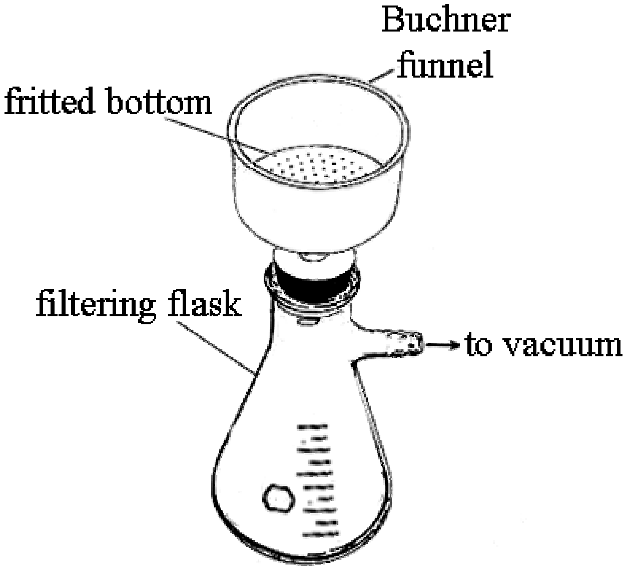


7. Finally, this is what the paper will look like when it is fully opened.



2) Vacuum Filtration (suction filtration)

Vacuum filtration is by far the most widely used and most important form of filtration, and its use in the modern lab is practically a must have and indispensable tool. Vacuum filtration is a process where a vacuum is applied to the filtering flask in order to pull the solution being filtered through the filter at a much faster rate. Unlike gravity filtration, vacuum filtration is very quick and virtually spontaneous in its rate of filtration, as the suction of the vacuum quickly pulls the liquid through the filter. The most common apparatus for use in vacuum filtration is a piece of equipment called a "Buchner" funnel, named after a famous chemist, and a vacuum source. A Buchner funnel is composed of a specially designed porcelain, plastic, or glass funnel, that contains a fritted flat bottom filled with tiny holes, as illustrated in the following illustration. The vacuum source can be either a standard vacuum pump, which are readily available, or a hand operated mechanical pump. Note: Buchner funnels often come in two pieces, the upper piece is the funnel and fritted bottom, and the bottom piece is usually the stem, and rubber stopper. To carryout a vacuum filtration process, simply pour the liquid you wish to filter into the Buchner funnel, and allow the vacuum to suck all the liquid through the filter. Once the filtration process is complete, you can simply vacuum dry your filtered-off mass, see section 2f for vacuum drying techniques.



Classic setup for vacuum filtration utilizing a Buchner funnel and a filtering flask. The Buchner funnel can either be plastic, porcelain, or glass.

Unlike gravity filtration, the filter paper used in vacuum filtration has a diameter only slightly smaller then the diameter of the Buchner funnel. This filter paper is placed on the inside of the Buchner funnel above the fritted bottom.

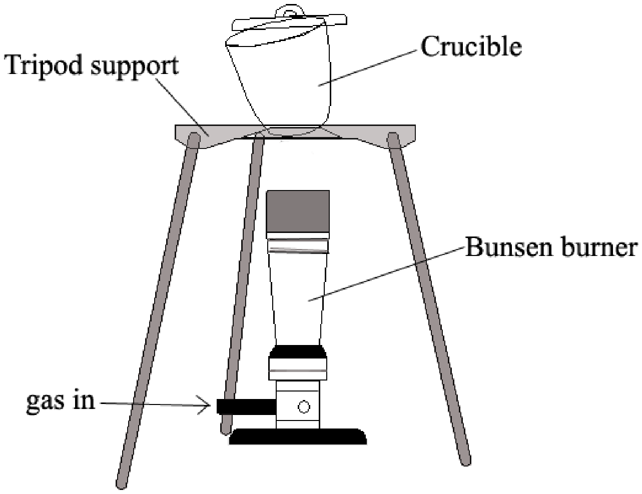
General Laboratory Techniques: Methods of heating

Heating is one of the most widely utilized tools in the general lab. Heat is used to stimulate a chemical reaction, boil a reaction mixture or solution, or to drive-off liquids as seen in distillation. The most important forms of heating in the general lab are discussed in the following:

1) Free flame

Free flames refer to Bunsen burners or alcohol lamps. Both utilize an open flame and can be used for general heating purposes. The pros of using free flames are that solutions and chemicals can be heated rapidly; however, the drawbacks to free flames are flammable liquids and their vapors. Heating combustible liquids with a free flame can be dangerous, as looming vapors can ignite. Other drawbacks to using free flames are that they don’t often heat mixtures or chemicals evenly, and the free flames can over-heat certain chemicals leading to dangerous situations such as dangerous decompositions resulting in toxic fumes, or just plain decomposition of the desired product.

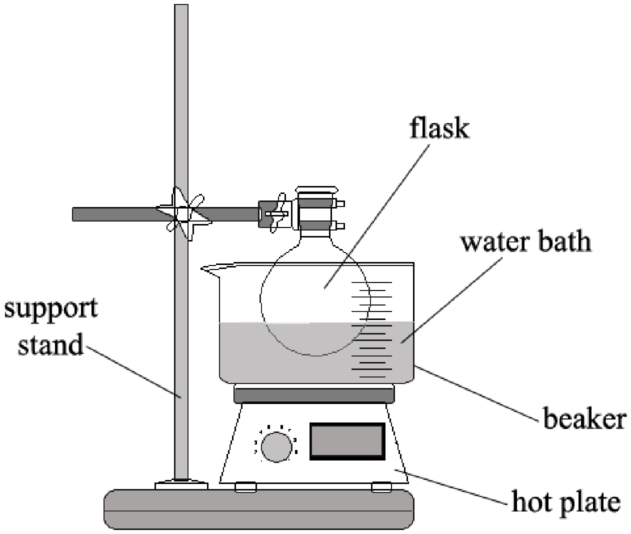
In using free flame devices such as Bunsen burners or alcohol lamps, the fuel is relatively cheap, owing to another pro for using free flames. When using Bunsen burners, simple natural gas or propane or even butane can be used. In the lab, natural gas outlets are readily available, and for home and school use, simple store bought propane tanks or butane tanks can be used as the fuel source.



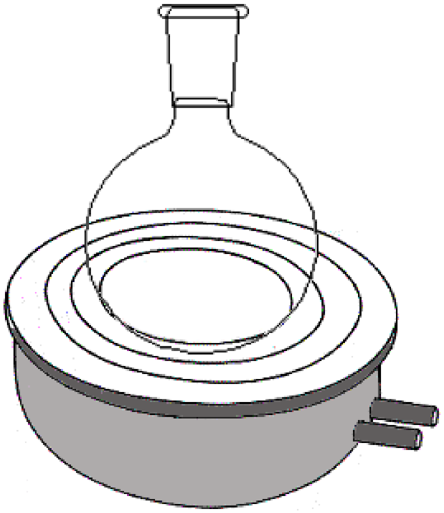
Common laboratory Bunsen burner with support stand.

2) Steam bath, or water bath

Steam baths, or water baths are used primarily to heat reaction vessels such as flasks to temperatures ranging from 50 to 125 Celsius. The beneficial aspect of steam baths are that they provide even heating without the dangers of forming blind spots. Blind spots occur when there is uneven heating. Blind spots can cause decomposition of a chemical compound, or can lead to unwanted chemical reactions due to poor and uneven heating. Another advantage steam baths or water baths have, is they eliminate the risk of igniting flammable fumes or the like. Steam baths, or water baths can be made using beakers or other heatable containers. The only draw back to steam baths or water baths is that they cannot heat to temperatures higher then 125 Celsius.



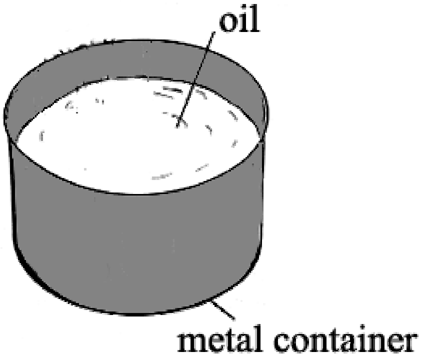
A classic setup for water bath heating. The hot plate is used to heat the water. Note: Notice how the flask is suspended in the water bath, and is not touching the bottom of the beaker.



A common steam bath. A steam bath is composed of a water container, and a heating element (not illustrated). The heating element boils the water, and the steam, is what actually heats the vessel. The vessel rests upon a system of rings that can be removed to accommodate larger vessels.

3) Oil bath

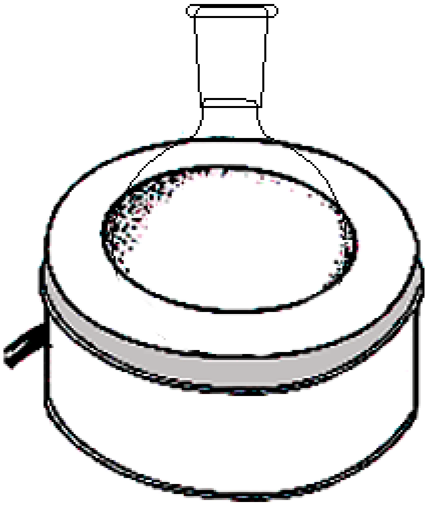
Oil baths are used in an identical manner as water baths, with the water simply being replaced by a non-volatile oil, such as a cooking oil, or crude oil compound. Oil baths are very useful for heating liquids and reaction mixtures to temperatures ranging from 125 to 350 Celsius. The only drawbacks to oil baths are they are slow to heat and cool, and they leave a greasy film on the flask being used, making it an annoyance for clean up.



A typical container for heating a flask with oil. The flask should be immersed only 1/3 of the way into the oil. Note: glass containers for holding the oil should be avoided.

4) Electric Heating Mantles

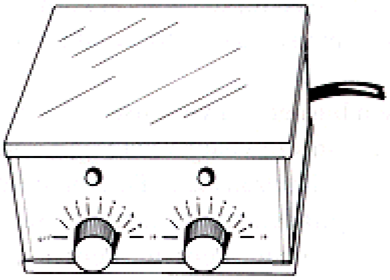
Heating mantles are very common for the use of heating flasks and other apparatus. Heating mantles are just like hot plates, except their shaped circular to accommodate round bottom flasks, which they are predominantly designed for. Heating mantles are very simple to operate, and come with variable temperature controllers. Heating mantles can be used to heat round bottom flasks and apparatus ranging from 50 milliliters up to 12 liters in capacity.



A typical heating mantle.

5) Hot Plates

Hot plates are by far the most common tool for heating in the general lab, and they are used extensively in heating flasks and beakers, and other flat bottom glassware or containers. Hot plates work in the same manner as heating mantles, except hot plates contain a flat smooth heating surface, usually composed of aluminum. Hot plates come in many different sizes, and some come with magnetic stirring devices built in.



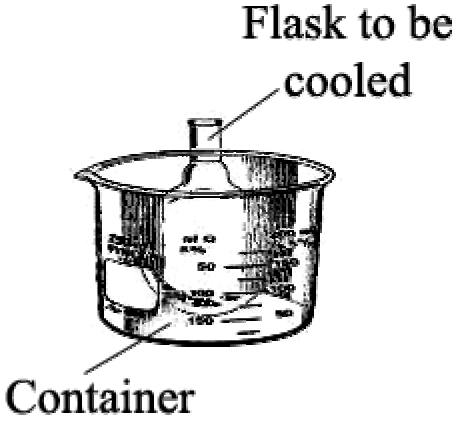
A typical hot plate. Most hot plates come doubled as hot plates and magnetic stirrers.

Methods of Cooling

As you will find in many chemical reactions, heat is often evolved; therefore, methods of cooling must be applied in order to maintain proper reaction conditions. Excessive heat build up during chemical reactions can lead to dangerous conditions including violent decomposition of a chemical or mixture, the evolution of harmful fumes or vapors, or the decomposition or loss of your desired product

When forming a method of cooling, one utilizes a large outer container, from which the cooling agent is then placed there into. For example, ice is the most common cooling agent, so when its used to form an ice bath, the bath, or container is usually three to four times the volume of your flask or reaction apparatus. If for example, your using a 1-liter flask to contain your chemicals or your reaction mixture, a 3-liter to 4-liter container should be used to house this 1-liter flask. Note: before adding the ice or cooling agent, make sure the flask or apparatus to be cooled is situated and seated inside the cooling vessel/container, as adding the ice first, will make it difficult to place the flask there into properly. Then when filling the ice bath with ice or other cooling agent, make sure to fill the ice bath all the way up. In short, if your using a 1-liter flask, it should be perfectly situated in a 3 to 4 liter cooling bath, and the ice or cooling agent, when placed there into, should submerge your 1-liter flask by at least 2/3 of the way up. In other words, 80% of the total height of your 1-liter flask should be totally covered or submerged in ice.

Note: In some cases your desired flask to be cooled may actually displace your cooling media (cold water, or ice water) before hand, or as the cooling media such as ice melts causing the flask to float and possible tip over. To alleviate this potential, Lead rings which fit around the necks of the flasks can be used to weight them down and keep them from tipping over.

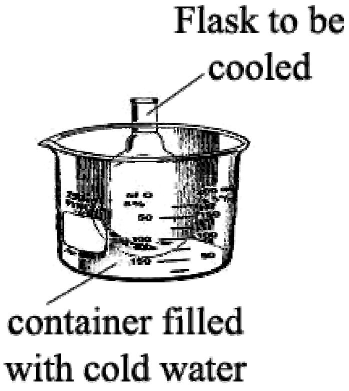


A typical setup for cooling a flask or reaction mixture. The container is filled with cooling agent.

Now, the five most common methods of cooling a flask or reaction apparatus will be discussed in the following:

1) Cold water bath

In some cases, cooling a flask can be done by the use of simple old-fashioned cold water. Cold-water baths are used to cool and maintain the flasks temperature from 15 to 60 Celsius. Note in some cases, during the chemical reaction or during the cooling process, the water will have to be quickly removed, and then replaced with fresh cold water, in order to maintain the proper temperature.



2) Ice water bath

Ice water baths are used in the same circumstances at plain old cold-water baths; however, they are generally used when the flasks temperature is to be kept below 50 Celsius. In forming ice water baths, the water to ice by volume should be about 50/50. Ice water baths are generally used to cool the flask to 15 to 40 Celsius.

3) Standard ice bath

Ice baths are the most common method of cooling flasks during reactions and other operations. Ice baths are generally used when cooling the flask to 0 to 25 Celsius is desired. For best results, the ice should be pulverized to allow for maximum surface area; unfortunately, a draw back to doing this can result in faster melting of the ice; therefore, small cubes of ice about half inch by half inch should be used to maximize surface area and allow the ice to adhere to the walls of the flask. Always remember to place your flask into the cooling container before adding the ice. During the cooling process, the ice will have to be replaced periodically, and this can be a pain. Note: for long term cooling and storage of a flask not involved in a chemical reaction, you can place the ice bath into a freezer for prolonged periods.

4) Salt/ice bath

The salt/ice bath is a modified version of the standard ice bath, and utilizes both ice and salt. In doing so, a cooling temperature of –10 to 5 Celsius can be maintained for short to moderate periods of time. Salt/ice baths can be easily prepared by simply placing the ice into your cooling container, after your desired flask has been placed, and then filling the container with crushed ice in the usual manner as for preparing an ice bath; thereafter, finely grained salt is added to the ice by sprinkling it over the ice. The ratio of ice to salt by weight should be about 90:10, meaning 90% ice, and 10% salt. As with standard ice baths, the cooling media may have to be replaced periodically, and this can be a pain. The most common salt used in salt/ice baths is common table salt (sodium chloride), but other salts can be used; for example, the salts of potassium chloride, magnesium chloride, and calcium chloride can be used as well. These salts generally produce cooling temperatures from –30 to 5 Celsius for short to moderate cooling periods.

5) Dry ice/acetone bath

Dry ice baths are very common in the modern lab, and can be used to achieve cooling temperatures of –70 to –10 Celsius. Because dry ice is so volatile, meaning it evaporates readily when exposed to air, the dry ice is often slurried with a solvent such as acetone, which preserves its cooling power for longer durations of time. As dry ice is readily available and inexpensive, it makes for a perfect cooling media. The drawbacks however, are the fact that it may over cool your flask, causing the contents therein to solidify or freeze. Therefore, dry ice/ acetone slurries are commonly used where low temperatures in general are required, such as maintaining a very low boiling liquid in its liquid state. Acetone isn't the only solvent that can be used, and other liquids, such as ethanol, ethyl acetate, or ether can be used. To create a dry ice/acetone bath, firmly secure your flask into the cooling container as with previous methods, and then pour in acetone (or other solvent) to the point where the cooling container is filled about half way. Thereafter, add in pulverized chunks of dry ice to completely surround the flask to be cooled and maximize surface area. The ratio of solvent to dry ice ranges from 50:50 (acetone to dry ice) for moderate to long cooling durations, to 70:30 (acetone to dry ice), for short term cooling operations. The advantage of using dry ice/solvent slurries is that the dry ice is readily replaced once it evaporates during the operation.

Cooling tricks of the trade

In some cases you will be able cool your flask by the use of a refrigerator and/or freezer. For long periods of time, a refrigerator/freezer can be used with great success. However, it is not wise to use a refrigerator for cooling if a chemical reaction is taking place in your flask, or if there are gases being evolved from your flask.

Recrystallization, and solid product recovery

Recrystallization

Recrystallization is the process of separating a dissolved solid product from a solvent solution. A solvent solution could be a previous extract from a reaction mixture, plant, ect., or a direct reaction mixture itself, or any type of solution containing a dissolved solid there into. A solution is what you get when a solid is dissolved into a liquid. For example, if we dissolved table salt into a glass of tap water, we would get a saline solution of sodium chloride in water. Now, in order to carryout a recrystallization we use three major methods of recovering the dissolved solid product, and these will be discussed below:

1) General recrystallization utilizing heat only

General recrystallization is a crucial tool used in chemistry to purify a non-heat sensitive solid product. Recrystallization sometimes follows the extraction process, which was briefly explained earlier. Unlike the extraction process however, recrystallization involves removing the chosen solvent to recover the dissolved product there into. In recrystallization, the idea is to remove the chosen solvent to recover a dry solid product. To do this, heat is applied to the solution in order to boil-off the chosen solvent. As the solvent is evaporated/boiled-off and removed, the solution containing the dissolved product becomes more and more concentrated. The more concentrated the solution gets, the closer you get to recrystallization. In recrystallization the goal is to remove enough solvent from the solution so as to form a "super saturated" solution. A super saturated solution is what happens when the dissolved product is more soluble in hot solution rather then in cold. What this means is, when we take a solution containing a dissolved solid, and we heat this solution to the point where its solvent begins to boil and evaporate-off, the solution becomes more and more concentrated. As such, the dissolved product becomes more soluble in the hot solution; however, when the heat is removed, and the solution is allowed to cool, the excess crystals in the super saturated solution have no solvent to go to as it was removed by evaporation/boiling, so they separate out as fine crystals. Once a few crystals form, a whole mess of crystals then grow off of them; thus, a recrystallization process has been successful. The precipitated crystals are then removed by filtration, and the remaining liquid solution is then heated to a boil again to restart the concentration process over again. This process is continued until the bulk of the dissolved solid has recrystallized and precipitated out, and the bulk of the solvent has been removed. Note: super saturated solutions are often called "mother liquors".

Working example of recrystallization using heat only

To use a working example of recrystallization using heat only, we will use a compound called ammonium chloride as the desired product, and water as the solvent.

1. Into a suitable beaker or flask, add and dissolve 100 grams of ammonium chloride into 400 milliliters of tap water.
2. Then place your flask or beaker onto a hot plate, and then heat the solution to 100 Celsius.
3. When the solution begins to boil, continue to boil the solution until you see a few small crystals floating on the surface of the solution.
4. When you see a few crystals of ammonium chloride floating on the surface of the boiling solution, remove the heat source and allow the solution to cool to room temperature. Note: you will begin to notice crystals separating out long before the solution is cooled to room temperature.
5. Once the solution has pretty much cooled to room temperature, or a few degrees above room temperature, filter-off all the precipitated crystals. At this point, you will notice that the total volume of the original solution has drastically changed.
6. Now, place the filtered solution back into your flask or beaker, and then heat the solution to 100 Celsius once again.
7. Allow the solution to boil, until you see a few tiny crystals floating on the surface of the solution.
8. When you see a few tiny crystals floating on the surface of the solution, remove the heat source, and allow the mixture to cool to room temperature as before. Note: you will begin to notice crystals separating out long before the solution is cooled to room temperature.
9. Once the solution has cooled to room temperature or near it, filter-off the precipitated crystals, using the same filter as for the first crop of precipitated crystals, and then place the filtered solution, which will have a reduced volume again, back into your flask or beaker.
10. At this point, your solution will have been reduced by at least 2/3rds in total volume. Continue the recrystallization process by heating the solution for a third time to 100 Celsius to boil-off the water once again.
11. Now, for a third time, when you see tiny crystals floating on the surface of the solution, remove the heat source and allow the solution to cool to room temperature for a third time. Note: you will begin to notice crystals separating out long before the solution is cooled to room temperature as before.
12. Finally, filter-off these crystals using the same filter as the pervious two. By this point the bulk of the ammonium chloride should have been recovered, and the bulk of the solution will have been removed.

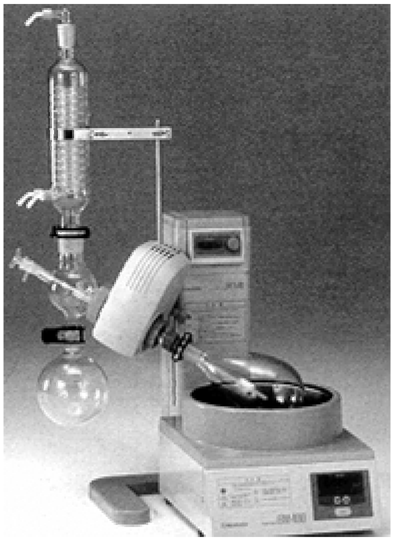
Note: in many cases, water will not be the solvent employed, and hence, you will want to use a distillation apparatus to boil off the solvent, and at the same time, recover the solvent. When using water, you can simply allow it to boil-off and dissipate in to the air, but when using solvents such as methylene chloride or ether, you will probably want to collect the solvent by using distillation instead of just allowing the vapors to boil away.

2) Recrystallization using seed crystals

Seed crystals are often used to induce a crystallization of a dissolved solid. To carryout a seed crystal process, take your solution containing the dissolved solid, and place it in the open, and allow the solvent to evaporate naturally. As the solvent evaporates the solution will become more and more concentrated. However, because a super saturated solution cannot form unless the mixtures boiled, as previously discussed, the solution will come to a point where its maximum solubility is achieved. When this point is reached, the crystals will only separate out slowly as the solvent evaporates from then on. In order to speed up the formation of crystals, we would employ a seed crystal. To do so, we simply take a couple of small dried crystals of the desired product, that we would get from a chemical supplier, and we place these seed crystals into the solution. In doing so, the crystals will begin to grow and will get larger and larger as the evaporation of the solvent continues. In most cases, seed crystals are primarily used to induce recrystallization from aqueous solutions and not organic solvents such as ethers, methylene chloride or others.

3) Recovering the product through low heat and vacuum

In most modern labs, the recrystallization process by heat or seed crystal is primarily reserved for aqueous solutions, and not organic mixtures. Organic mixtures on the other hand, such as solutions containing the solvents ether, methylene chloride, ethyl acetate, or other common organic solvents are placed into devices called rotary evaporators to remove the solvent and recover the dissolved product. A rotary evaporator, as illustrated below, is a special device, which utilizes low heat and high vacuum in order to remove the solvent. When a vacuum is applied to a system containing a mixture, the boiling point of the solvent is drastically reduced. With lower boiling points comes lower heating temperature needed to drive off the solvent. In doing so, low heat can be used to drive-off the solvent, and collect the dissolved solid product from the solution. By using rotary evaporators, the solvents can be driven-off at faster rates drastically speeding up the process of separation. Also, with lower heats being used, heat and other sensitive products can be recovered without danger of decomposition. Note: liquids have decreased boiling points with decreased pressures, and higher boiling points with increasing pressures (water boils at 100 Celsius at sea level, but boils at 93 Celsius above 15,000 feet, and so on).



A classic rotary evaporator. The solution to be evaporated is placed into a diagonal flask, which is partially submerged in a water bath. A vacuum is applied, and the water bath is gently heated to the desired temperature. As the solvent is removed, it collects in the receiver flask located on the bottom left of the illustration.

Washing liquids

Liquids are not always needed to be washed, but every so often, they will need a quick washing or scrubbing. To wash a liquid, simply place the liquid into a separatory funnel, and then add in water or another solvent. Note: the water or liquid being used for the washing should not dissolve the liquid to be washed, and when added to the separatory funnel should form a two-phase mixture. Thereafter, shake the separatory funnel vigorously for a few moments, and then allow the separatory funnel to stand to allow the two-phase mixture to form—similar to an extraction process. Now, if the liquid your using to wash is the upper layer, you will need to drain-off the bottom layer first, before draining off the upper layer, and then you will have to place the first drained-off portion back into the same separatory funnel after draining off the upper layer. If the liquid you're using to wash is the lower layer, then simply drain it off, leaving the upper layer in the separatory funnel. Repeat this process three or four more times to thoroughly wash your liquid.

Washing solids using non-vacuum techniques

The method of washing solids via non-vacuum techniques is accomplished by placing the solid product to be washed into a beaker or other suitable container, and then adding in an excess of water or solvent. Note: the solid to be washed should be insoluble in the water or solvent used. The ratio of solid product to liquid should be about 1 part solid product to 10 parts liquid. Thereafter, stir the entire mixture for a few moments, and then allow the mixture to stand for several minutes. During this standing time, the insoluble solid product will slowly settle to the bottom. After the solid product has settled to the bottom, most of the water or solvent will be above the settled solid product. Now is the tricky part, the excess liquid should be carefully drained-off by titling the beaker or container at an angle so that the liquid slowly drains out without the insoluble solid being removed. Thereafter, more water or solid is then added and the process is repeated again. This repeating can be as many times as possible but usually requires about 6 or 7 repeats before the solid is totally washed.

Washing solids using vacuum techniques

Solids are easily washed by vacuum filtration by placing the solids to be washed on a piece of filter paper placed above the fritted bottom of a Buchner funnel. Thereafter, a clean liquid, such as water or any desired pure solvent is then poured in, in moderate size portions, over the solids in the Buchner funnel. After each pouring, the liquid is sucked through the filter paper, thereby cleaning the solids in the Buchner funnel. This process of pouring can be repeated a many times as desired. Note: the liquid your using to clean and wash the solids in the Buchner funnel should not be able to dissolve the solids being washed.
If washing a product after it has been filtered from a liquid or mixture, simply pour the filtered liquid used during the vacuum filtration process, back into the Buchner funnel. This pouring of the filtered liquid back into the Buchner funnel can be repeated as many times as necessary. Each time, the liquid will be sucked through the filter via the vacuum, and will act to wash the insoluble material. In most cases, the filtered liquid is only poured back into the Buchner funnel a few times, before a fresh

liquid is used. The fresh liquid is usually water when dealing with inorganic and organic substances. Note: of coarse water cannot be used to wash water reactive materials. When using a fresh liquid such as water, the liquid is poured in portions, and allowed to be sucked through the filter via the vacuum thereby washing the insoluble mass.

Drying solids

Solids can be dried using four main techniques. These four techniques include using an oven to dry the solids, using a vacuum to dry the solids, using airflow to dry the solids, or using a hot plate to heat and dry the solids. When using an oven, the solids are merely placed into an oven, and heated to a temperature that will dry the solids. Ovens are usually used to dry solids that are moist with water, and solids moist with flammable solvents should not be dried using ovens.
Vacuum drying is the most common method of drying many types of solids and works in the same manner as vacuum filtration. In vacuum drying, the solids to be dried are placed onto a piece of filter paper inside a Buchner funnel, and a strong vacuum is applied. As the suction of the vacuum sucks air through the filter paper, the airflow will dry the solids as a result. Vacuum drying is the most common form of drying solids in the lab.
One method of drying solids is to place the solids onto a shallow pan or tray, and allowing them to air dry. To speed up the air drying process, a small fan can be used to blow air over the solids, thereby drying them at a moderate rate. This form of drying is useful in drying large quantities of heat sensitive solids.
The final form of drying solids is to use direct heat from a hot plate. To dry solids in this manner, the solids are simply placed into a beaker or similar container, and then heated directly using a hot plate. This form of drying can be used to dry solids quickly, but is limited to solids that are not heat sensitive. Heat sensitive solids cannot be dried in this manner.

Drying liquids to remove water

Drying liquids containing traces of water can be accomplished by mixing the liquid to be dried with a drying agent. This form of drying is commonly used after an extraction process, whereby the combined extract portions are freed from traces of water by using a drying agent. Reaction mixtures and other liquids can also be dried and freed from traces of water by using drying agents. To dry a combined extract portion, reaction mixture or any other liquid, simply place a small amount of a drying agent into the mixture, and then stir the mixture for a few minutes. During the stirring process, the drying agent will absorb any traces of water that may be present. Thereafter, the drying agent, which will be insoluble in the mixture, can be removed by filtration.

The following are the most commonly used drying agents used in the modern lab:

1) Anhydrous sodium sulfate

Anhydrous sodium sulfate is one of the most widely used drying agents used in the lab to dry organic liquids to remove tracers of water. It is very inexpensive, relatively inert, can be recycled over and over again, and it has a very high absorption of water as it can form a decahdyrate. The sodium sulfate can be recycled after its use by placing it into a crucible and heating it to about 200 Celsius using a Bunsen burner.

2) Anhydrous magnesium sulfate

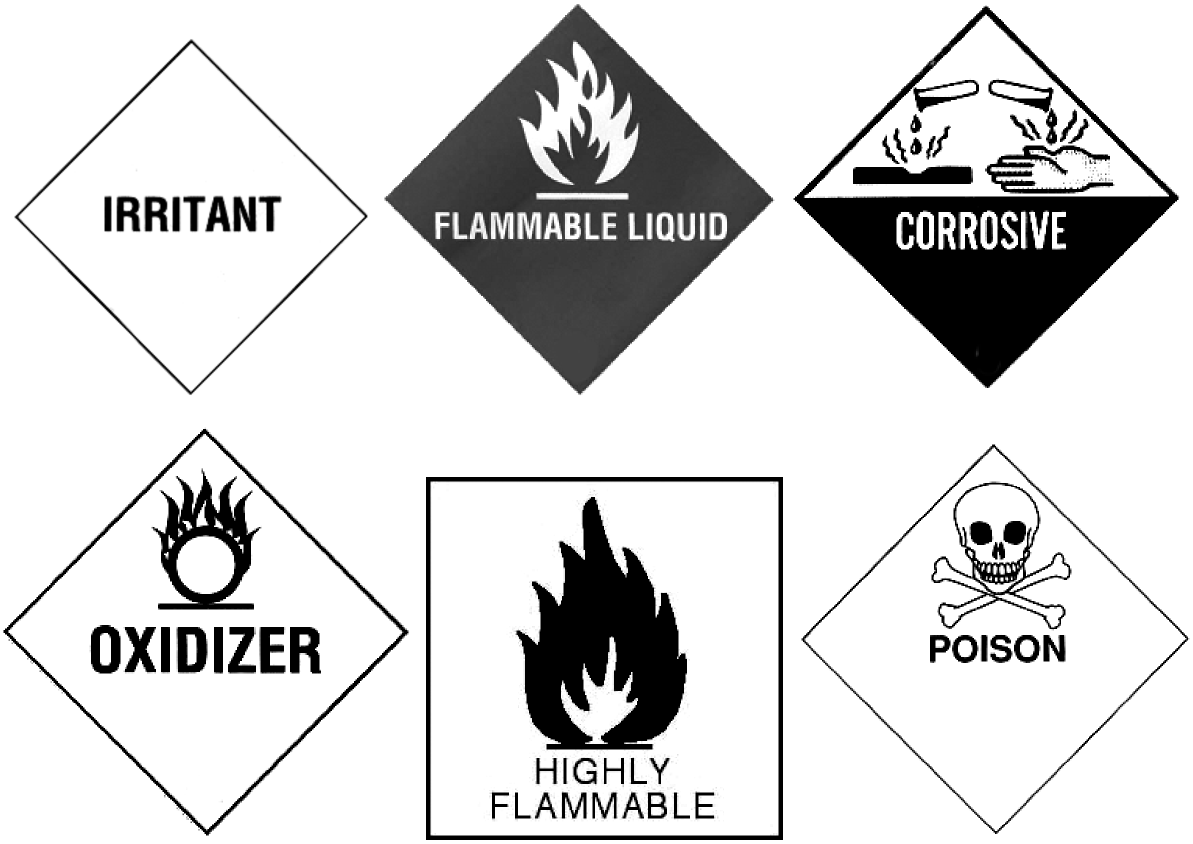
Anhydrous magnesium sulfate is another very common and inexpensive drying agent used to dry organic liquids to remove traces of water. Anhydrous magnesium sulfate has very similar properties and next to sodium sulfate, is the second most common drying agent used in the lab. One advantage that anhydrous magnesium sulfate has over sodium sulfate, is that it has a higher affinity for water then sodium sulfate.

3) Calcium chloride

Calcium chloride is the third most common drying agent used to dry organic liquids to remove traces of water. Calcium chloride has a very high affinity for water, and its affinity for water is greater then that of sodium or magnesium sulfates. Calcium chloride is very effective, and it removes traces of water readily and rapidly; however, the draw back to calcium chloride is its reactivity towards substances and organic liquids. Calcium chloride cannot be used to dry organic liquids containing ammonia, amines, alcohols, acids, and tetrahydrofurans.

Laboratory safety

Now that you have a brief understanding of basic chemistry and general laboratory techniques, now is the time to familiarize yourself with laboratory and chemical safety. Laboratory and chemical safety is crucial in order to maintain proper organization and discipline when dealing with laboratory procedures, chemicals, and general techniques. When dealing with laboratory procedures and chemicals, the best form of safety is to avoid and prevent accidents before they happen. **Under all circumstances, eye protection and gloves should be used at all times when dealing with chemicals. Eye protection includes eye goggles that form a complete seal around the eyes and face, and eye glasses alone should not be used for eye protection**. **Most gloves worn in the lab are called "Nitrile" gloves, which are chemically resistant to almost all compounds.** When dealing with chemicals, you need to familiarize yourself with chemical warning hazard signs as illustrated below:



Common laboratory chemical warning signs.

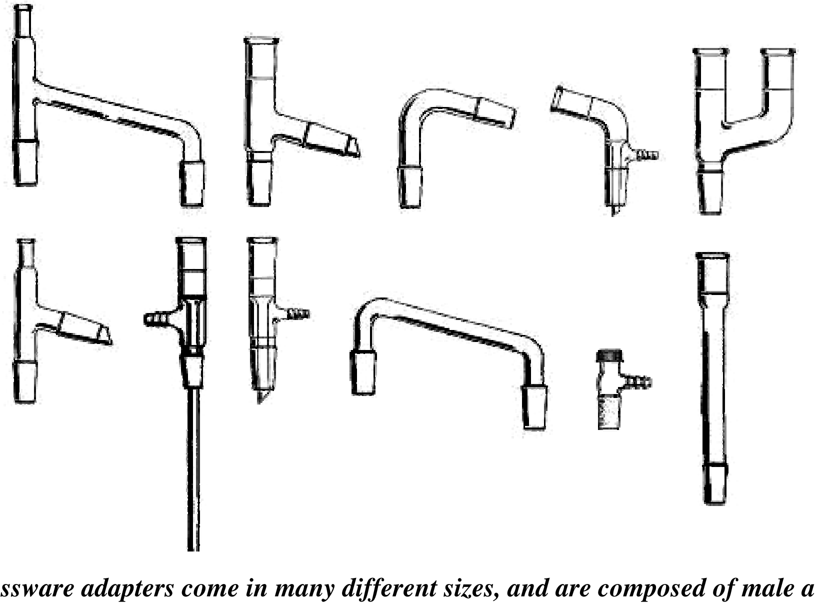
For the general dealing with and handling of chemicals, the following checkpoints should be observed:
1) Always wear safety goggles at all times. Clothing, laboratory apparatus, and equipment can be replaced, but your eyes cannot. Do not use contact lenses or eyeglasses alone as a substitute for safety goggles. Always remember, if you get chemicals into your eyes under any circumstance, immediately flush your eyes with large excessive volumes of fresh cold tap water.
2) If you spill a chemical on your body, immediately remove any clothing that comes into contact, and flush any exposed skin with large amounts of cold tap water. It should be noted that most chemicals are only dangerous if they are allowed to linger on the clothing or skin, so take action immediately. Note: even concentrated sulfuric acid spilled on your clothing or skin is harmless as long as you take action immediately. Waiting around and the acid will dissolve through your clothing and begin to burn your skin.
3) In the case of fire or possible explosion, always remember to save yourself first. Trying to be a hero by battling the blaze or trying to prevent an explosion can get you hurt. You can keep fire extinguishers in arms reach, and you can keep an adequate water source within reach, but do not attempt to take on the accident by yourself.
4) For simple accidents such as acid spills, keep baking soda in arms reach. Baking soda can be spread upon acid spills to neutralize them.
5) When working with flammable solvents and liquids, make sure that all sources of ignition are suppressed. In the event of a fire you should carefully yet calmly move away from the burning area. If the fire is small and appears to be fightable, go ahead and try to extinguish the flames; if however, the fire is fast and progressive, evacuate the area. As before, do not attempt to be a hero.
6) It is of great importance that you do not eat or drink anything while handling chemicals or carrying out laboratory processes. Food and beverages can become contaminated without your knowledge, and by accident.
7) By all means, never attempt to taste chemicals or attempt to place chemicals on your body in anyway.

8) Never smell chemicals directly by sticking your nose up to beakers, flasks, or any other containers. Chemicals in open containers can be sampled by wafting the vapors with your hand to your nose, while keeping a safe distance from the open container.
9) Make sure not to rub your eyes or scratch your body while wearing gloves and handling chemicals. Traces of chemicals on your gloves or bare hands can be transferred to other parts of your body.
10) It should be noted that breathing and/or skin contact with small concentrations or small amounts of noxious liquids, mixtures, or any other substances does not pose an immediate danger, but you should avoid contact with any potentially noxious chemical under all circumstances. Toxic chemicals should only be handled by personnel who know what they are doing. In any case, toxic substances should be handled with great care and attention. Monitor for any potential leaks or spills.
11) Always handle volatile solvents and liquids in well-ventilated areas, and avoid breathing fumes. Flammable liquids like ether are highly volatile, and their fumes can fill rooms and travel great distances in short periods of time. Also, try to avoid breathing the vapors of flammable solvents as they can get you buzzed and make you feel dizzy, decreasing your bodies ability to focus and concentrate on what your doing.
12) When working with chemicals, always try to wear inexpensive clothing such as work clothing since the possibility of an accident is always present. Inexpensive clothing can be damaged during the accident, but replaceable. When working with chemicals, laboratory coats and/or aprons are readily available and can be worn over existing clothing.
13) Do not wear loose clothing, sandals, or casual clothing while working with chemicals. Obviously, shorts, skirts, blouses, or any other clothing that leaves large areas of skin unprotected should not be worn when handling chemicals.
14) Never experiment with unfamiliar chemicals by heating them, mixing them, or treating them with any other chemicals, or any other sort of interaction. Never heat unfamiliar chemicals as decomposition forming toxic fumes may result.
15) When carrying out chemical reactions, always make sure you know what you are doing, or have knowledge about what you are doing before you do it. Playing around with chemicals can lead to the generation of toxic fumes, explosions, or fire hazards.

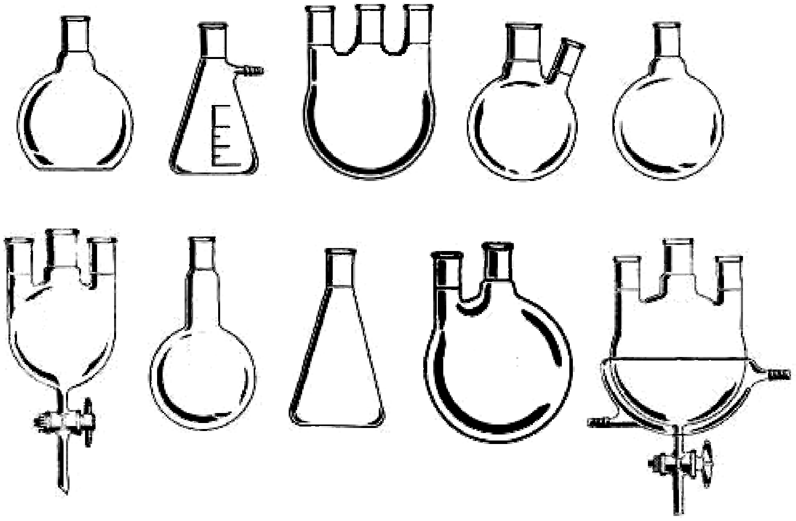
Now the basic general chemistry and general laboratory techniques just included will not prepare you for the experiments in this book, as many of the experiments in this book contain radical formulas, dangerous chemicals, and great advanced apparatus that has to be custom made, and can be very expensive.

Laboratory glassware

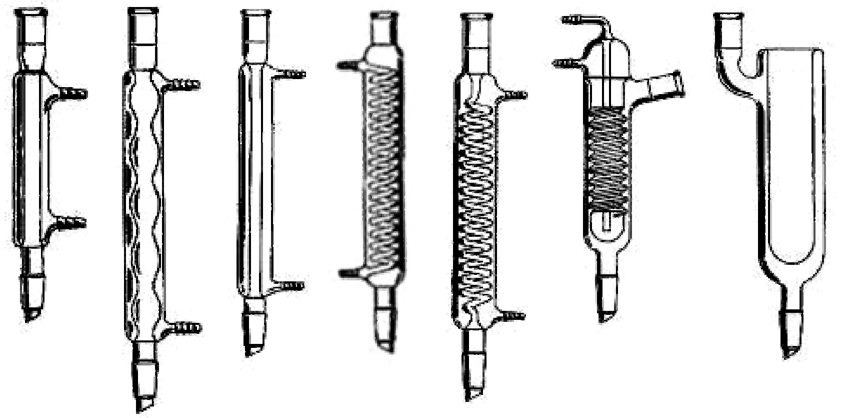
Laboratory glassware are essential when carrying out extractions, chemical reactions, or chemical processes. Laboratory glassware comes in many different sizes and forms, all of which are made of heat resistant glass called “Pyrex”, or “Kimax”. This heat resistant glassware can be heated to temperatures of up to 500 Celsius without breakage, cracking, or scarring. Because laboratory glassware varies in size and type, the glassware ranges in price, and some glassware can be quit expensive. In any sense, its time you get familiar with general laboratory glassware. Laboratory glassware is divided into five main categories, and these categories include 1) adapters, 2) flasks, 3) condensers, 4) general lab glass, and 5) specialty apparatus.



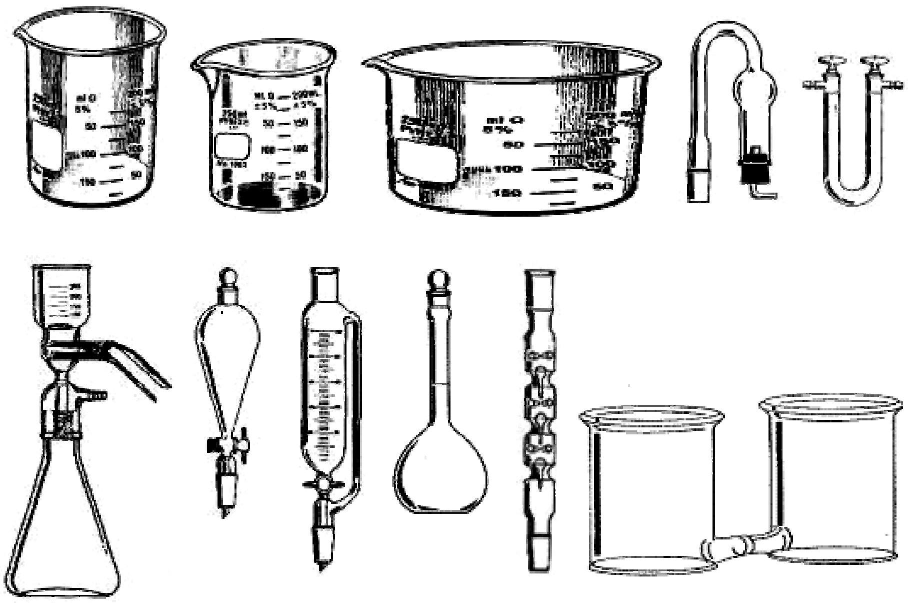
Adapters. Laboratory glassware adapters come in many different sizes, and are composed of male and female glass joints. The joints range in size from 10/30, to 19/22, to 24/40. The adapters have male and female joints that are composed of etched glass surfaces, which form an airtight seal when placed male to female. Adapters range in price from $25.00 to $150.00.



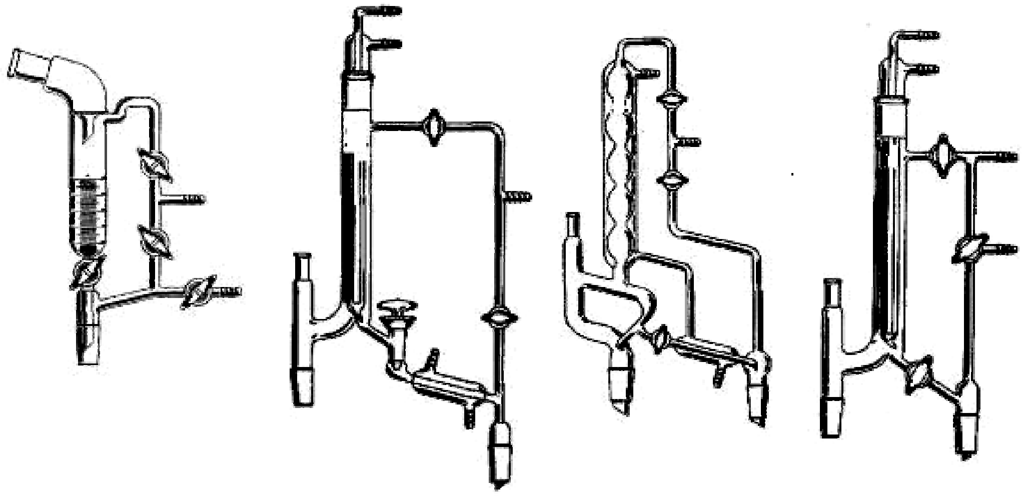
Glass flasks. As with adapters, many flasks in the chemical laboratory are composed of etched joints ranging from 10/30 to 24/40. Flasks range in price from $15.00 to $300.



Laboratory glassware condensers. Like adapters and flasks, most condensers in the modern lab have etched glass joints. Condensers range in price from $45.00 to $500.



Common laboratory glassware. Other forms of common laboratory glassware are test tubes, which are not shown in the illustration. Some common laboratory glassware contains etched glass joints as seen in adapters, flasks, and condensers. Common laboratory glassware ranges from $1.00, to $250.



Advanced laboratory glassware. These types of glassware can be very expensive and range in price from $500 to $1000.

Even though laboratory glassware can be heated to temperatures ranging from 100 Celsius to 500 Celsius. Laboratory glassware should not be subjected to cool temperatures after heating, as crackage can occur.

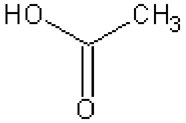
Laboratory equipment

General laboratory equipment ranges from hot plates, Bunsen burners, to ovens. Other types of laboratory equipment include vacuum pumps, oil baths, and rotary evaporators. Laboratory equipment ranges from $30 to $6000. Rotary evaporators can cost as much as $6000. Advanced laboratory equipment includes HPGC, and NMR systems, HPGC stands for high performance gas chromatography, and NMR stands for Nuclear Magnetic Resonance. High performance gas chromatography can range from $6000 to $15,000 and are used to identify chemical compounds in parts per million. High performance gas chromatography is widely used in modern labs, and is practically indispensable. High Performance gas chromatography is widely used by research scientists and analytical scientists to identify chemical compounds.

In some of the experiments in this book, the apparatus is often made of nickel, and not heat resistant glassware like Pyrex or Kimax.

Section 2: Intermediates, Reagents, and Solvents

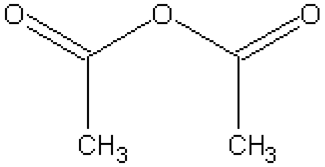
Acetic acid, Glacial. *Pure acetic acid*



Acetic acid

Glacial acetic acid is a colorless liquid with a pungent odor. Glacial acetic acid is a corrosive liquid with a boiling point of 118 Celsius, and a melting point of 17 Celsius. Glacial acetic acid is a flammable liquid. It is an excellent solvent for many organic compounds, and is miscible with water, alcohol, glycerol, ether, and carbon tetrachloride. Glacial acetic acid weekly ionizes in water solutions. It can be made by the destructive distillation of wood, and subsequent condensation of the vapors (methanol is a by-product and hence must be separated by distillation). Glacial acetic acid is a common commercially available chemical. *Wear gloves when handling glacial acetic acid because it can cause skin irritation and possible skin burns.*

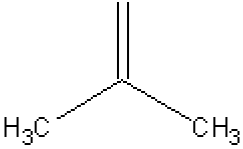
Acetic anhydride



Acetic anhydride

Acetic anhydride is a colorless liquid with a melting point of –73 Celsius, and a boiling point of 139 Celsius. The liquid is corrosive and has a strong acetic acid odor. When mixed with water, it slowly decomposes into acetic acid, and it reacts with alcohols forming acetates. Acetic anhydride is soluble in chloroform and ether. It is made by reacting sodium acetate with acetyl chloride or sulfuryl chloride. Acetic anhydride may produce irritation of the skin, so wear gloves when handling.

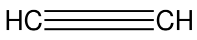
Acetone



Acetone

Acetone is a very volatile, highly flammable liquid with a rather characteristic odor, and a pungent, sweetish taste. It has a boiling point of 56 Celsius and a melting point of –94 Celsius. Acetone is miscible with water, alcohol, chloroform, ether, and most oils. Acetone will dissolve many plastics, and resins. Keep acetone away from plastic eyeglass frames, jewelry, pens and pencils, rayon stockings, and other rayon garments. It is mildly irritating, and fumes produce dizziness, and headaches. Use proper ventilation when handling acetone. Acetone is prepared by the oxidation of isopropyl alcohol with potassium dichromate, and on an industrial scale by the oxidation of cumene with air. Acetone is a very common and widely available solvent.

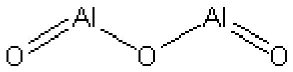
Acetylene gas



Acetylene gas

Acetylene gas forms a colorless with not an unpleasant odor when pure, but has a disagreeable odor when contaminated with impurities. The gas is toxic when inhaled. The compound has a melting point of –81 Celsius, and it is easily liquefied when cooled to 0 Celsius at 21.5 ATM's. The gas burns with brilliance in air with a sooty flame, but when excess oxygen is added, it burns with a smokeless very hot flame. Acetylene forms explosive compounds with copper and silver when passed over copper or silver turnings. Acetylene is easily made by dripping water onto calcium carbide.

Alumina



Aluminum oxide

Aluminum oxide forms a white crystalline powder. The powder becomes insoluble in acids when heated to 800 Celsius. The crystalline powder is insoluble in water and all organic solvents. It is made by reacting aluminum chloride hexahydrate with sodium hydroxide, followed by filtration to recover the aluminum hydroxide, which is then dehydrated by roasting at 200 Celsius for several hours. Alumina is widely used in chromatography, and for filtration to remove impurities from reaction mixtures.

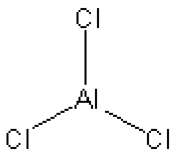
Aluminum foil

Aluminum foil is a shinny silvery-white foil that is very flexible, tearable, and easy to cut. It does not melt when heated as it forms an oxide layer. Aluminum foil is highly reactive to hydrochloric acid, sulfuric acid and sodium and potassium hydroxides liberating large volumes of hydrogen gas.

Aluminum, powdered

Powdered aluminum forms a silvery-white to grayish to grayish-black to black powder that is highly flammable and forms explosive mixtures with strong oxidizers. Powdered aluminum is readily available at numerous sources.

Aluminum chloride, Anhydrous



Aluminum chloride

Anhydrous aluminum chloride is white when pure, but is ordinarily a gray or yellowish powder, which fumes in air, and has a strong hydrogen chloride odor. Anhydrous aluminum chloride volatizes when heated in small quantities. It combines with water with explosive violence and liberation of much heat. Anhydrous aluminum chloride is very soluble in most organic solvents. Anhydrous aluminum chloride is a powerful catalyst for the halogenation of organic compounds. It is prepared by passing dry hydrogen chloride gas over finely divided aluminum.

Aluminum nitride

$$AlN$$

Aluminum nitride forms orthorhombic or hexagonal, bluish white crystals, with a melting point of 2200 Celsius. The compound has a faint odor of ammonia in moist air, and is hydrolyzed by water into aluminum hydroxide and ammonia. Aluminum nitride is a very hard substance that is only slightly less harder then boron carbide. Aluminum nitride is made by passing nitrogen over heated bauxite and coal. In the lab, aluminum nitride is formed by heating powdered aluminum in a stream of nitrogen gas under an inert atmosphere of argon.

Ammonia, anhydrous

$$NH_3$$

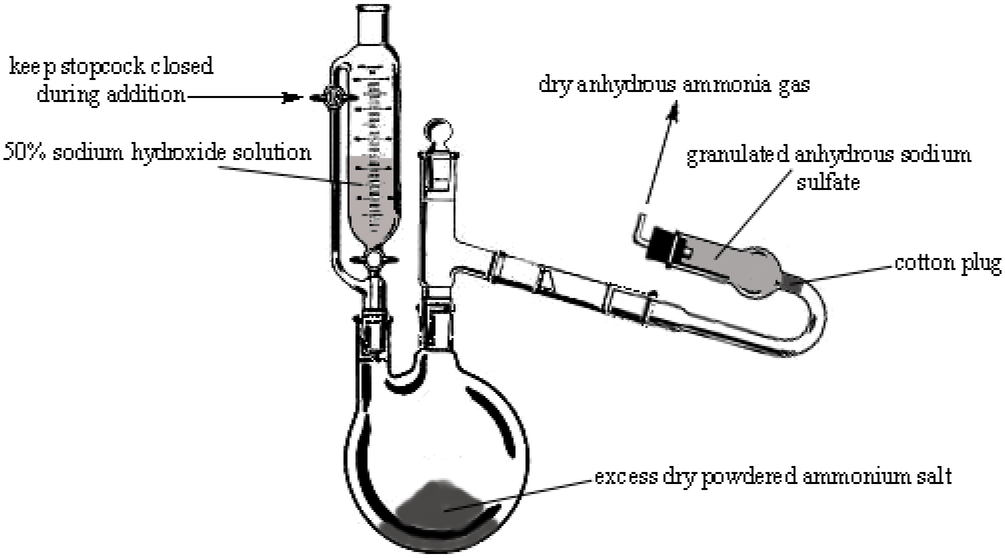
Anhydrous ammonia is a colorless gas with a very pungent odor. It has a melting point of -77 Celsius, and a boiling point of -33 Celsius. Ammonia is regarded as nonflammable, but mixtures with oxygen can ignite. Anhydrous ammonia is a corrosive alkaline gas that is very soluble in water. It is also soluble in alcohol, chloroform, and ether. Liquid ammonia is a good solvent for many elements and compounds. Commercial anhydrous ammonia is supplied in the form of a compressed gas in steel tanks, or in the liquid form supplied in steel tanks. Ammonia is also widely sold in water solutions. Anhydrous ammonia is the 4th largest chemical produced in the US. Anhydrous ammonia is a widely available commercial chemical. Anhydrous ammonia is prepared on an industrial scale from hydrogen and nitrogen at high pressure and temperature. The average ammonia plant produces 1000 tons of liquid ammonia per day.

Method of preparing anhydrous ammonia

Summary: Anhydrous ammonia can be prepared by treating an ammonium salt solution with a solution of sodium hydroxide. In the following procedure, ammonium chloride or ammonium sulfate is treated with sodium hydroxide to yield ammonia gas, which is then dried to yield the anhydrous gas. The ammonium chloride or sulfate can be prepared by neutralizing 10% ammonia (store bought ammonia) with hydrochloric or sulfuric acids, followed by recrystallization.

Hazards: Wear gloves when handling 50% sodium hydroxide. The alkaline solution may cause painful skin burns on prolonged exposure, and itching sensation on short exposure. Use proper ventilation when making ammonia gas, and avoid inhalation of the vapors.

Procedure: Place 156 grams of 10% ammonia into a flask and then rapidly add 100 grams of 35% hydrochloric acid, or 47 grams of 98% sulfuric acid while stirring the 10% ammonia solution. After the addition of the acid, continue stirring for ten minutes, and then recrystallize the ammonium salt from solution. Afterwards, place the dry recrystallized ammonium salt into an apparatus as illustrated below, and then prepare a sodium hydroxide solution by dissolving 38 grams of sodium hydroxide into 38 milliliters of water (much heat is produced when dissolving sodium hydroxide into water). After the sodium hydroxide solution has cooled to room temperature, add it the ammonium salt drop-wise over a period of about forty minutes. During the addition of the sodium hydroxide, ammonia gas will be steadily evolved.


keep stopcock closed during addition
50% sodium hydroxide solution
dry anhydrous ammonia gas
granulated anhydrous sodium sulfate
cotton plug
excess dry powdered ammonium salt

Apparatus for the preparation of anhydrous ammonia

20% aqueous ammonia solution

Prepare by dissolving anhydrous ammonia gas into 400 milliliters of water.

28 to 30% ammonia solution

28- 30% Ammonia can be prepared by dissolving 22 grams of ammonia gas into 48 milliliters of water. 28 – 30% ammonia is a highly irritating liquid, which evolves highly irritating vapors. Use proper ventilation when handling this substance. 28 – 30% ammonia solution is commercially available.

35% aqueous ammonia solution

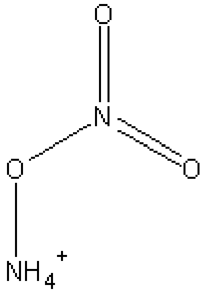
Prepare by dissolving 50 grams of anhydrous ammonia into 90 milliliters of water. Note: keep sealed in a glass bottle in a freezer until use.

Ammonia, liquid

$$NH_3$$

Liquid ammonia has a boiling point of –33 Celsius, and is a highly volatile and corrosive liquid, which readily volatizes forming a powerful and highly irritating vapor. It is easily made by liquefying anhydrous ammonia gas.

Ammonium nitrate



Ammonium nitrate

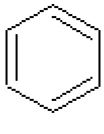
Ammonium nitrate forms odorless, transparent hygroscopic crystals or white granules. The crystals are very soluble in water with moderate solubility in alcohol. The crystals decompose when heated to 210 Celsius, forming water and nitrous oxide. Catalysts increase the formation of nitrous oxide. Ammonium nitrate is readily available commercially.

Argon

$$Ar$$

Argon forms a colorless, odorless, tasteless gas, which is totally inert to chemical attack; however, can form compounds with fluorine gas but only suitable conditions. The gas comprises about 1% of the earths atmosphere, and is obtained by the distillation of liquid air.

Benzene



Benzene

Benzene is a clear, colorless, volatile, highly flammable liquid. Benzene is toxic, and a carcinogen. It has a boiling point of 80 Celsius, and is miscible with alcohol, chloroform, ether, carbon disulfide, and carbon tetrachloride. It is insoluble in water. Keep benzene in tightly sealed glass bottles, and store in a cool area. Benzene is a common well-known solvent available commercially, but purchasing may be difficult due its EPA regulation. *Use proper ventilation when handling benzene, as inhalation of benzene vapors are hazardous.*

Boric anhydride

$$B_2O_3$$

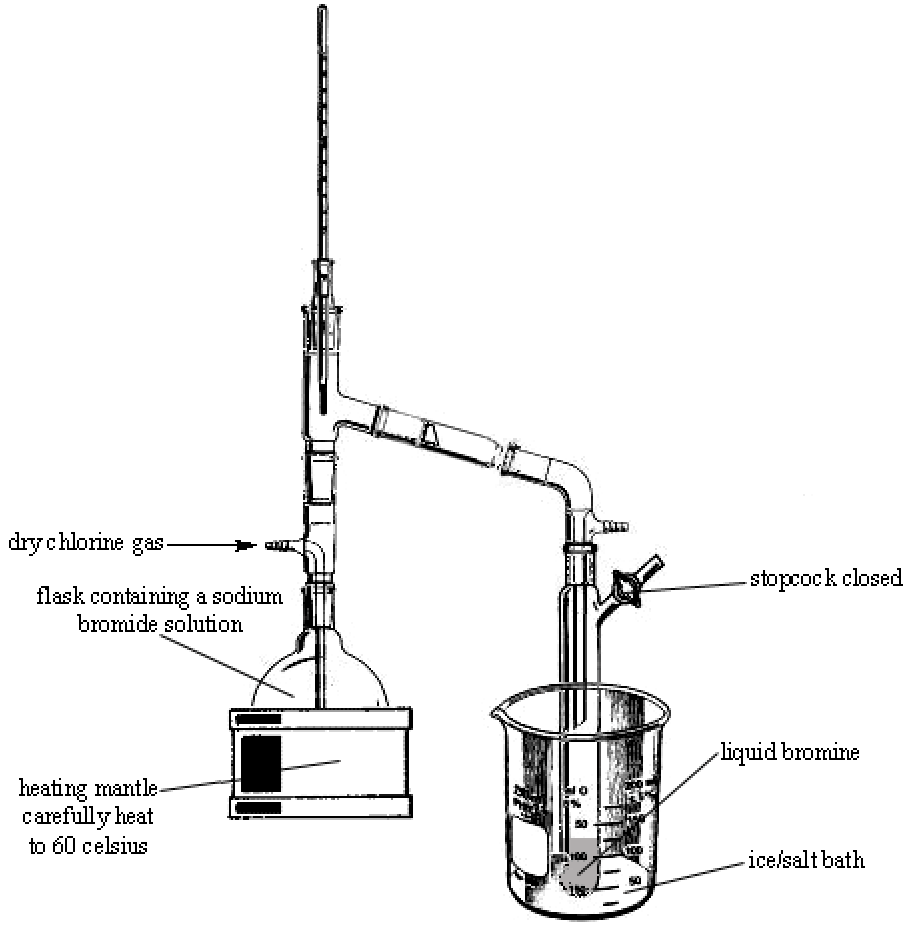
Boric anhydride forms colorless, brittle, semitransparent, hygroscopic, lumps, or hard white crystals. The crystals have a melting point of 450 Celsius. The crystals are relatively insoluble in water, but soluble in alcohol and glycerol. Boric anhydride can be prepared in the lab by heating dry powdered boric acid under mild vacuum in a sublimation apparatus.

Bromine

$$Br_2$$

Bromine

Bromine is a dark red, highly fuming liquid, which is very volatile. Its fumes are toxic, corrosive, and strongly irritating. Bromine has a melting point of -7 Celsius, and a boiling point of 59 Celsius. It is insoluble in water, but freely soluble in alcohol, ether, chloroform, and carbon disulfide. It is soluble in alkali bromide solutions. Bromine is less reactive then chlorine, but just as toxic. Keep bromine stored in glass stoppered bottles, and store in a cool place (refrigerator) away from sunlight. Bromine is prepared by passing chlorine gas into a solution of sodium bromide, and then simultaneously evaporating the bromine. The bromine vapors are then condensed. Bromine is commercially available but shipping regulations may restrict its sale.


dry chlorine gas
flask containing a sodium bromide solution
heating mantle carefully heat to 60 celsius
stopcock closed
liquid bromine
ice/salt bath

Apparatus for preparing bromine. The bromine should be re-distilled at 60 Celsius.

Carbon, activated

Activated carbon is also known as carbon black, and has a quasi-graphitic form of carbon made by heating animal bones, collected by the burning of methane with little oxygen, or by burning fats and oils. The compound forms a black powder that will burn under proper conditions, and wood charcoal contains activated carbon.

Charcoal

Charcoal powder forms a blackish grayish substance that is obtained by destructive distillation of wood. The powder is flammable, and is used in making gunpowder. Never burn charcoal in enclosed areas as toxic carbon monoxide is produced.

Carbon monoxide

$$CO$$

Carbon monoxide

Carbon monoxide is a colorless, odorless gas with a melting point of –205 Celsius, and a boiling point of –191 Celsius. It is highly poisonous and is an effective blood agent as it binds with oxygen carriers within the blood stream. The gas is flammable, and burns to carbon dioxide. Carbon monoxide is insoluble in water, but soluble in solutions of cuprous chloride in the presence of hydrochloric acid, and in concentrated ammonia solutions. The gas dissolves to moderate extents in ethyl acetate, chloroform, and acetic acid. Carbon monoxide is highly toxic and inhalation should be avoided under all circumstances. Use proper ventilation when handling. Carbon monoxide is a major by-product in the production of iron from blast furnaces, as it is produced upon reduction of iron ores with coke and lime. It is also produced as a product from incomplete combustion of natural gas.

Chloroform

$$CHCl_3$$

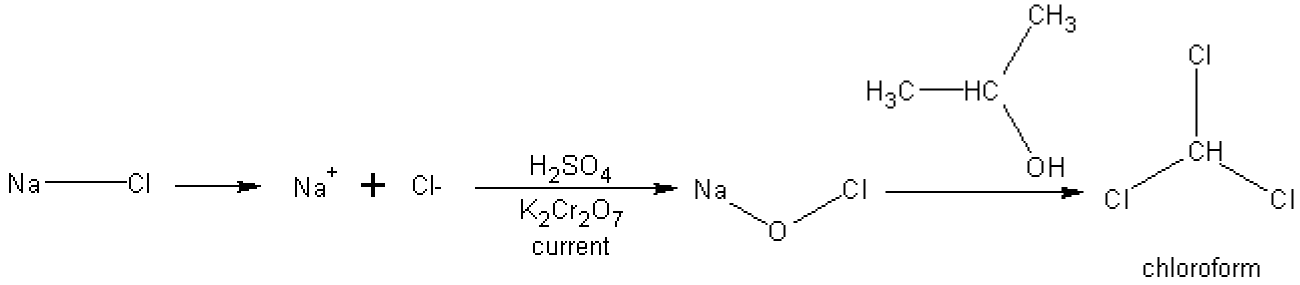
Chloroform is a highly refractive, nonflammable, heavy, very volatile, and sweet-tasting liquid with a peculiar odor. It has a boiling point of 62 Celsius, and a melting point of –64 Celsius. Chloroform forms a constant boiling mixture with alcohol containing 7% alcohol, and boiling at 59 Celsius. Commercial chloroform contains a very small amount of ethanol as stabilizer. It is insoluble in water, but miscible with alcohol, benzene, ether, petroleum ether, and carbon disulfide. Pure chloroform is light sensitive, so store in amber glass bottles in a cool place. Chloroform is a suspected light carcinogen, so use proper ventilation when handling. Over exposure to chloroform vapors causes dizziness, and headache. **Note:** Distilling mixtures containing chloroform mixed with one or more strong base (lithium, sodium, or potassium hydroxide) can result in explosion or violent reaction. Always neutralize any base, or extract the chloroform before distilling.

Method 1: Preparation of chloroform from Rubbing alcohol, potassium dichromate, pickling salt, and dilute sulfuric acid using an electrochemical cell

Materials:

1. 40 grams (1.4 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy addictives—just plain old rubbing alcohol)	5. 375 milliliters (12.6 fluid oz) of warm tap water
2. 148 grams (5.2 oz.) of pickling salt	6. 225 milliliters (7.6 fluid oz.) of benzene, toluene, or xylene
3. 1 gram of potassium dichromate (available on-line)	7. 5 milliliters of concentrated sulfuric acid (1 teaspoon) (plumbers grade or better)
4. 1 milliliter of 95% ethyl alcohol	8. 15 grams of anhydrous magnesium sulfate

Reaction summary: Chloroform can be prepared in a unusual manner by electrolyzing a solution of pickling salt and isopropyl alcohol in the presence of a small amount of sulfuric acid and potassium dichromate using graphite or lead electrodes. The process develops low yields of chloroform, and takes considerable amounts of time, but is effective at producing small amounts of chloroform. After the initial reaction, the mixture is extracted with benzene, toluene, or xylene, and the resulting solvent extracts are then combined, and then distilled to recover the chloroform.

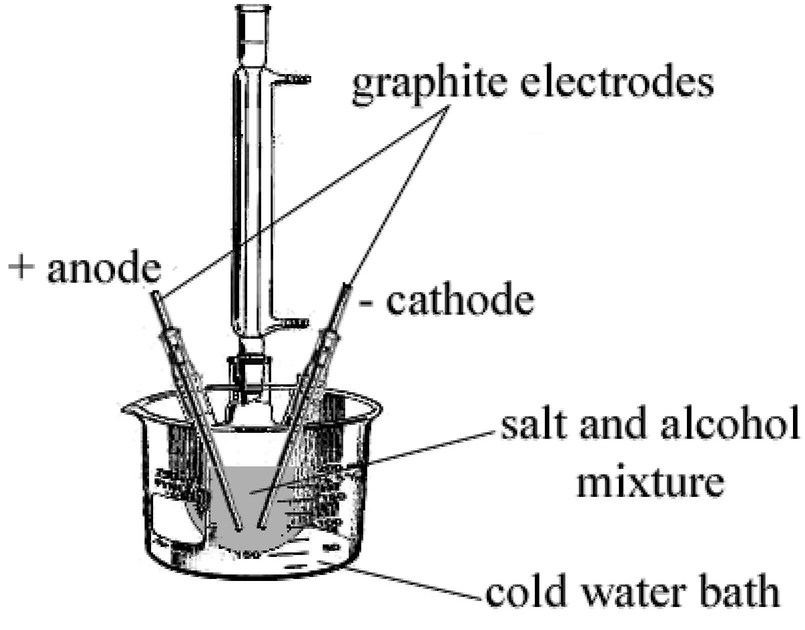


Hazards: Avoid breathing in the fumes of the reaction mixture. Use proper ventilation when carrying out this procedure.

Procedure: Into a suitable separatory funnel, place 40 grams (1.4 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy addictives—just plain old rubbing alcohol), followed by 20 to 30 grams (0.7 to 1 oz.) of pickling salt. Note: make sure the stopcock on the separatory funnel is closed before starting. Thereafter, stopper the separatory funnel, and then shake the entire funnel vigorously for about 5 minutes. Then allow the separatory funnel to stand (upright) for about 30 minutes. Thereafter, drain-off the bottom brine layer (and any excess salt), and thereafter, recover the upper isopropyl alcohol layer. Now, pour this isopropyl alcohol into a suitable container (see the following illustration for electrochemical cell make-up), and then add in 118 grams (4.1 oz.) of pickling salt, followed by 375 milliliters (12.6 fluid oz) of warm tap water, followed by 1 gram of potassium dichromate, and then followed by 5 milliliters of concentrated sulfuric acid. Thereafter, vigorously stir the entire mixture for about 30 minutes to dissolve all solids. At this point, you will end up with an orangey colored solution. Now, setup your electro chemical cell, and then electrolysize this mixture for 15 hours at 12 volt 6 amp current. Use a battery charger with this setting—these battery chargers are very common and availed in many stores. After 15 hours, unplug the battery charger, and then pour the entire mixture into a beaker, and then extract this entire mixture with three 75-milliliter portions (2.5 fluid oz. portions) of benzene, toluene, or xylene, and after the extraction process, combine all solvent portions, if not already done so. Thereafter, dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered dried solvent mixture into a distillation apparatus, and distill over the chloroform at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform into a clean fractional distillation apparatus and distill at 62 Celsius until no more chloroform passes over into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place.

Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

The electrolysis process



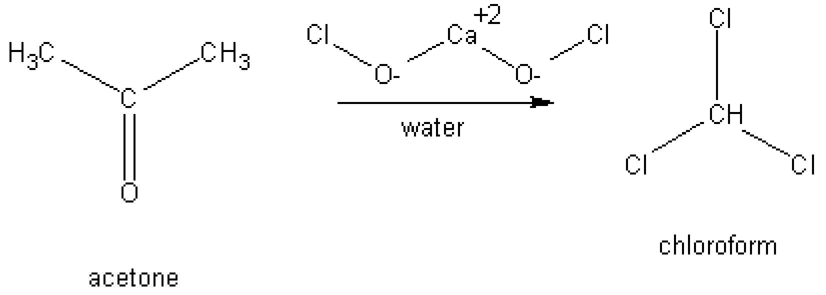
Electro chemical cell for the preparation of chloroform. This apparatus is a laboratory apparatus, and can be replaced with similar apparatus designs. The 3-neck flask shown in the illustration can be replaced with a large canning jar, by cutting three holes in the lid, or something of that nature. Other containers can be used, and you should use your imagination. Note: the red clamp of your battery charger goes on the positive anode, and the black clamp goes on the negative cathode. Graphite rods of various lengths and diameters are available on line for reasonable prices.

Method 2: Preparation of chloroform from acetone and bleaching powder

Materials:

1. 100 milliliters of tap water (3.4 fluid oz.).	3. 1 milliliter of 95% ethyl alcohol (see entry).
2. 100 grams of acetone (3.5 oz.). Readily available in any hardware store.	4. 300 milliliters (10.1 fluid oz.) of benzene, toluene, or xylene. Toluene and xylene should be available at most hardware stores.
2. 1181 grams (2.6 pounds) of 65 to 70% calcium hypochlorite (bleaching powder) (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs). Can also be found online at many pool and spa chemical suppliers and can be purchased without hassles.	5. 15 grams of anhydrous magnesium sulfate (obtained by heating Epsom salt in an oven, microwave, or Bunsen burner).

Reaction summary: Chloroform is prepared by reacting acetone with calcium hypochlorite (bleaching powder), and then extracting the mixture with benzene, toluene, or xylene. After extraction, the solvent/chloroform mixture is then distilled to collect the chloroform, which is then re-distilled. After collecting the chloroform after re-distillation, it is mixed with a small amount of 95% ethanol to act as a stabilizing agent.

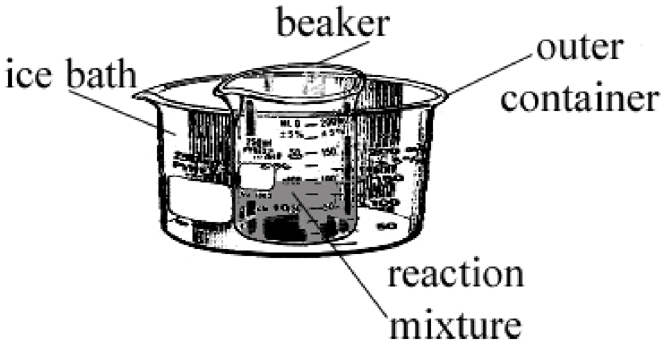


Hazards: Extinguish all flames before using acetone, which is highly volatile and flammable. ***Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concentrated sulfuric acid; explosions will result.*** Chloroform inhalation should be avoided, but is not threatening in mild conditions. Benzene, toluene, and xylene are suspected carcinogens so avoid prolonged exposure to fumes and vapors.

Procedure: Place 100 milliliters of tap water (3.4 fluid oz.) and 100 grams of acetone (3.5 oz.) into a beaker or any suitable container, and then cool this mixture to 0 Celsius using a standard ice bath. Thereafter, slowly add in small portions, 1181 grams (2.6 pounds) of 65 to 70% calcium hypochlorite (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs) over a period of 1 hour while stirring the acetone solution and maintaining its temperature at 0 Celsius. During the addition of the calcium hypochlorite, rapidly stir the acetone/water mixture, and maintain its temperature below 20 Celsius. After the addition of the 65 to 70% calcium hypochlorite, continue to stir the reaction mixture at 0 Celsius for an additional thirty minutes. Afterwards, stop stirring and then extract the reaction mixture with four 75-milliliter portions (four 2.5 fluid oz. portions) of benzene, toluene, or xylene. After extraction, combine all four portions (if not already done so), and then dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered dried solvent portion into a distillation apparatus (as illustrated below) and then distill at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform in a clean fractional distillation apparatus (as illustrated below) and distill at 62 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place.

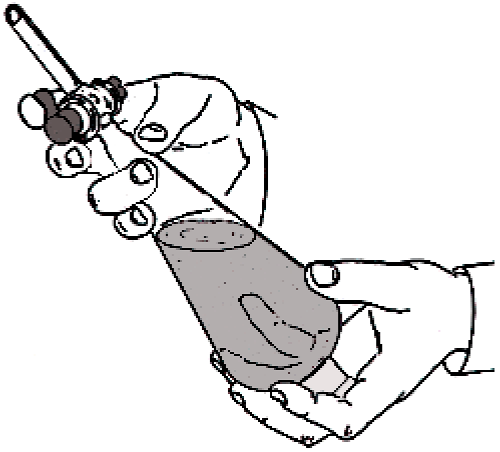
Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

Step 1: Initial reaction of calcium hypochlorite with acetone.



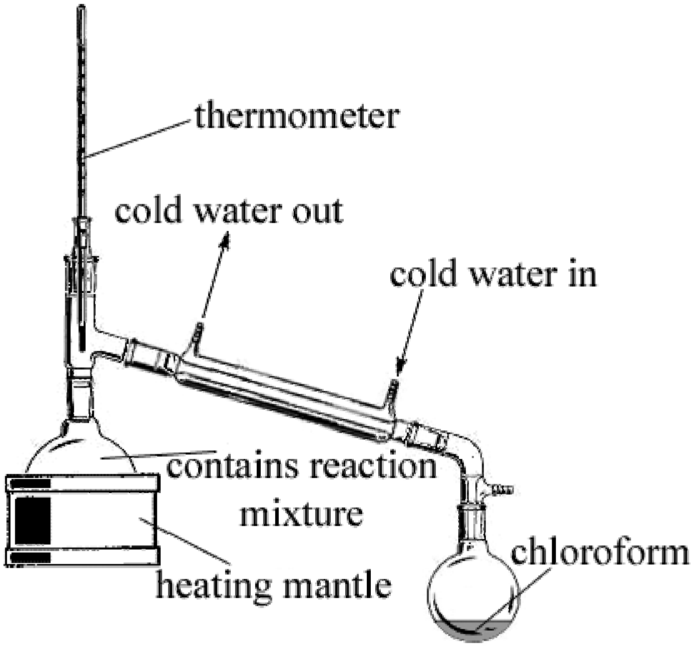
Set-up with ice bath for cooling the reaction mixture. The beaker can be re-placed with a flask or other suitable container. However, the container should not be made of plastic or other polymer that may corrode or dissolve by the acetone or chloroform. The outer container for use as the ice bath, can be glass, plastic, metal, or any other similar container.

Step 2: Extraction process



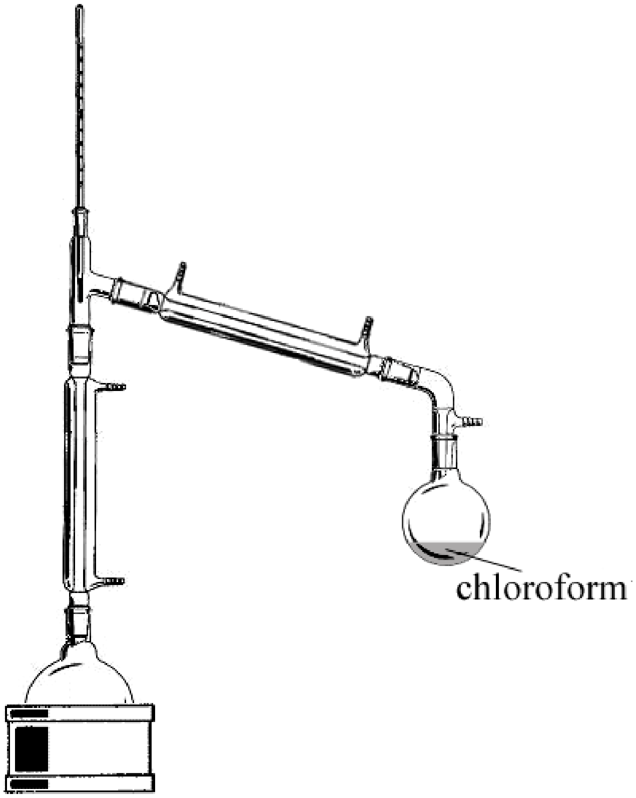
Extract the reaction mixture with benzene, toluene, or xylene.

Step 3: Distillation process to recover the chloroform



Distillation apparatus for collecting the chloroform. The heating mantle can be replaced with a Bunsen burner, but the flame should not come into direct contact with the distillation flask (as bumping and foaming may result). The chloroform should be re-distilled using a fractional distillation apparatus for quality and purity.

Step 4: Fractional distillation apparatus for purifying the chloroform



Fractional distillation apparatus for the fractional distillation of chloroform. The heating mantle can be replaced with a Bunsen burner, but the flame should not touch the glass. A hot plate or stovetop can also be used as a heat source if desired.

Chlorine gas

$$Cl_2$$

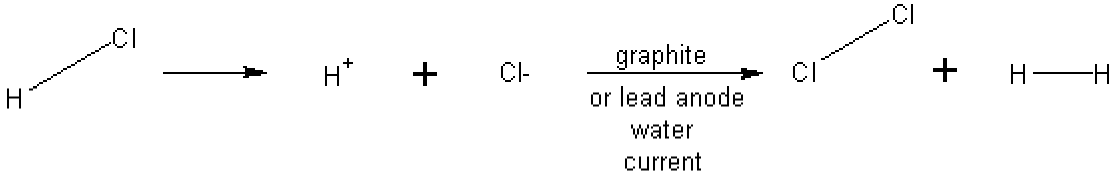
Chlorine gas is a yellow gas with a suffocating, and strongly irritating odor. It has a melting point of –101 Celsius, and a boiling point of –34 Celsius. Chlorine is sold as a compressed gas in steel cylinders. It is insoluble in water and not very soluble in alcohol, but soluble in dry benzene, and toluene. Chlorine combines readily with all elements except the noble gases, hydrogen, oxygen, and nitrogen. Chlorine does not occur naturally, but occurs in combined form as chlorides. It occurs in nature (in the form of chlorides) as sodium chloride, potassium chloride, and magnesium chloride. Many finely divided metals will burn in a chlorine atmosphere. Chlorine is a toxic gas, which can be fatal if inhaled for prolonged periods of time. Inhalation of mild quantities of chlorine causes nose and throat irritation followed by excessive mucous congestion. Chlorine is a corrosive gas, which will react with many metals on contact. It is a strong oxidizer and is capable of oxidizing a great many inorganic compounds. Chlorine will explode in contact with hydrogen if direct sunlight is present. Chlorine should be protected from sunlight. It is prepared on an industrial scale from the electrolyses of sodium chloride brine in a system called the chloro-alkali process (sodium hydroxide is a useful by-product). It can be prepared in the lab by reacting hydrochloric acid with calcium hypochlorite or any other strong oxidizer

Method 1: Preparation of chlorine gas from dilute hydrochloric acid utilizing an open cell

Materials:

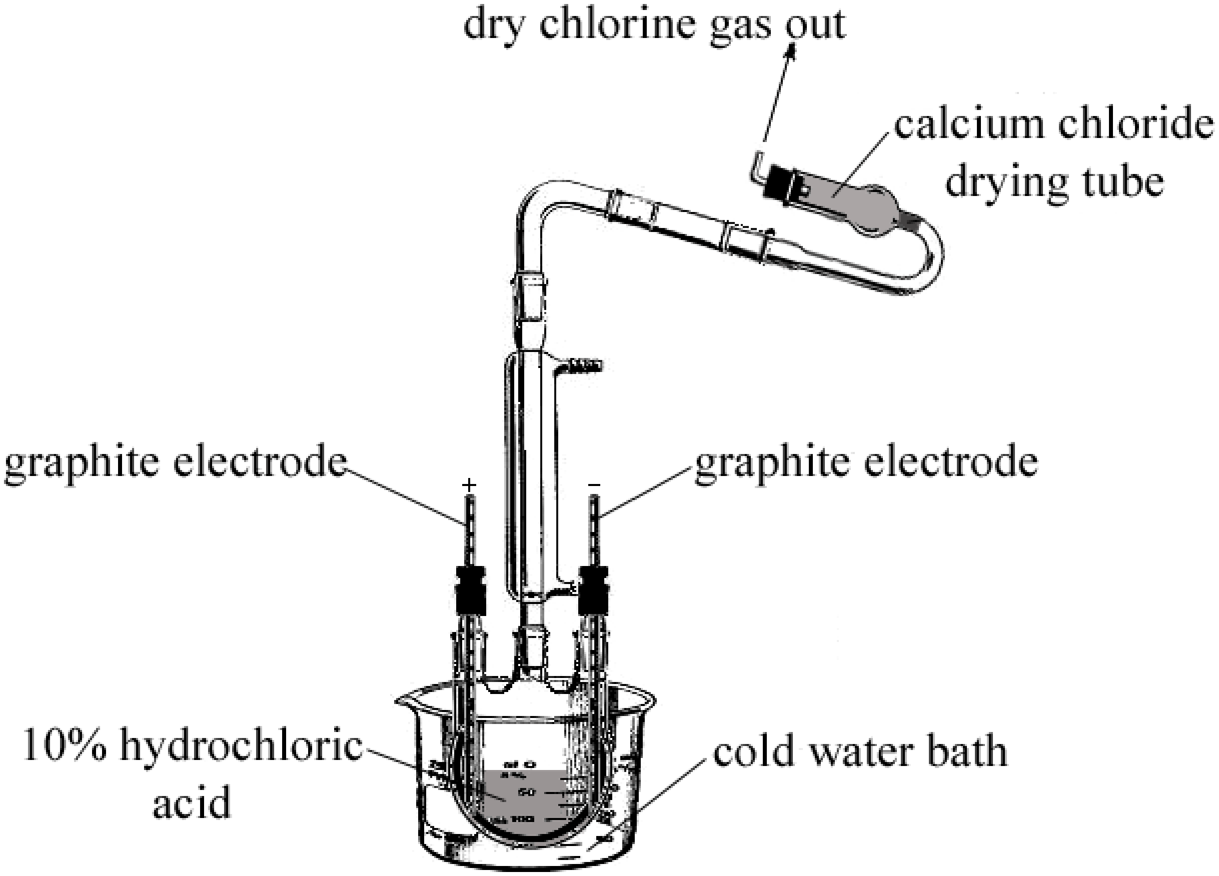
1. 400 milliliters (13.5 fluid oz.) of cold water	2. 100 milliliters (3.3 fluid oz.) of concentrated hydrochloric acid or concentrated Muriatic acid

Reaction summary: Chlorine gas is readily prepared by the electrolysis of hydrochloric acid. The reaction generates heat, so the flask or container should be submerged in a cold-water bath.

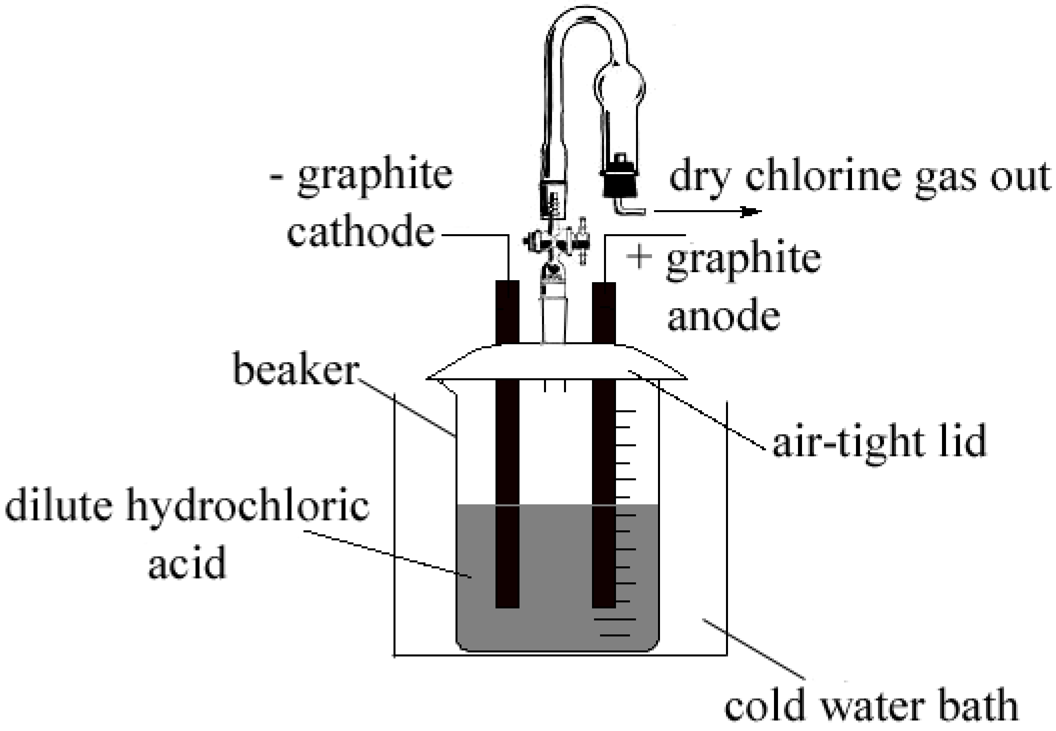


Hazards: Use proper ventilation when handing chlorine gas, and avoid contact with direct sunlight.

Procedure: Into either of the apparatus listed below, place 400 milliliters (13.5 fluid oz.) of cold water, followed by 100 milliliters (3.3 fluid oz.) of concentrated hydrochloric acid or concentrated Muriatic acid. Thereafter, turn on the power supply and run the cell for about 6 to 7 hours at 2 to 12 volt at 2 to 6 amp. The amperage determines how long the process will take. Higher amperage is preferred. Note: This process will produce about 31 grams of chlorine gas. Note: the graphite electrodes can be replaced with lead if desired. NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that the electrolysis of hydrochloric acid utilizing an open cell is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.



Apparatus for the electrolysis of hydrochloric acid. Warning: If the apparatus is exposed to direct sunlight, an explosion will occur—as hydrogen gas is also liberated (hydrogen gas at the negative electrode, and the chlorine gas at the positive anode). Chlorine explodes when mixed with hydrogen and then exposed to sunlight (only when exposed to sunlight). The detonation propagates downwards, so there is no immediate danger from fragments. As a reminder, the electrolysis of hydrochloric acid is perfectly safe as long as the apparatus is protected from direct sunlight, magnesium light, halogen lamps, or UV lamps (cover all windows, ect.). Note: the black clamp of your battery charge goes on the (-) electrode, and the red clamp of the battery charger goes on the (+) electrode. Use a current of 6 volt 2 amp or what ever works best for you. If using 6 volt 2 amp, it should take about 2 hours for the chlorine evolution to cease.



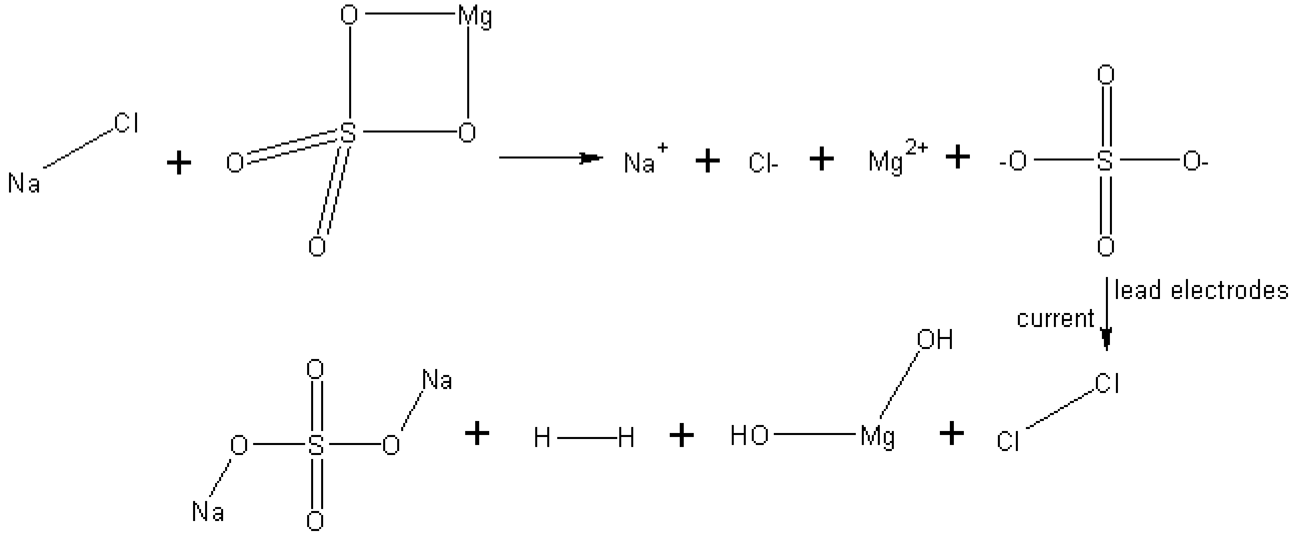
Apparatus for the preparation of chlorine gas. Keep the apparatus away from direct sunlight, magnesium light, or UV light. Note: the entire apparatus should be airtight, 100%

Preparation of chlorine gas from Epsom salt, and pickling salt utilizing an open cell

Materials:

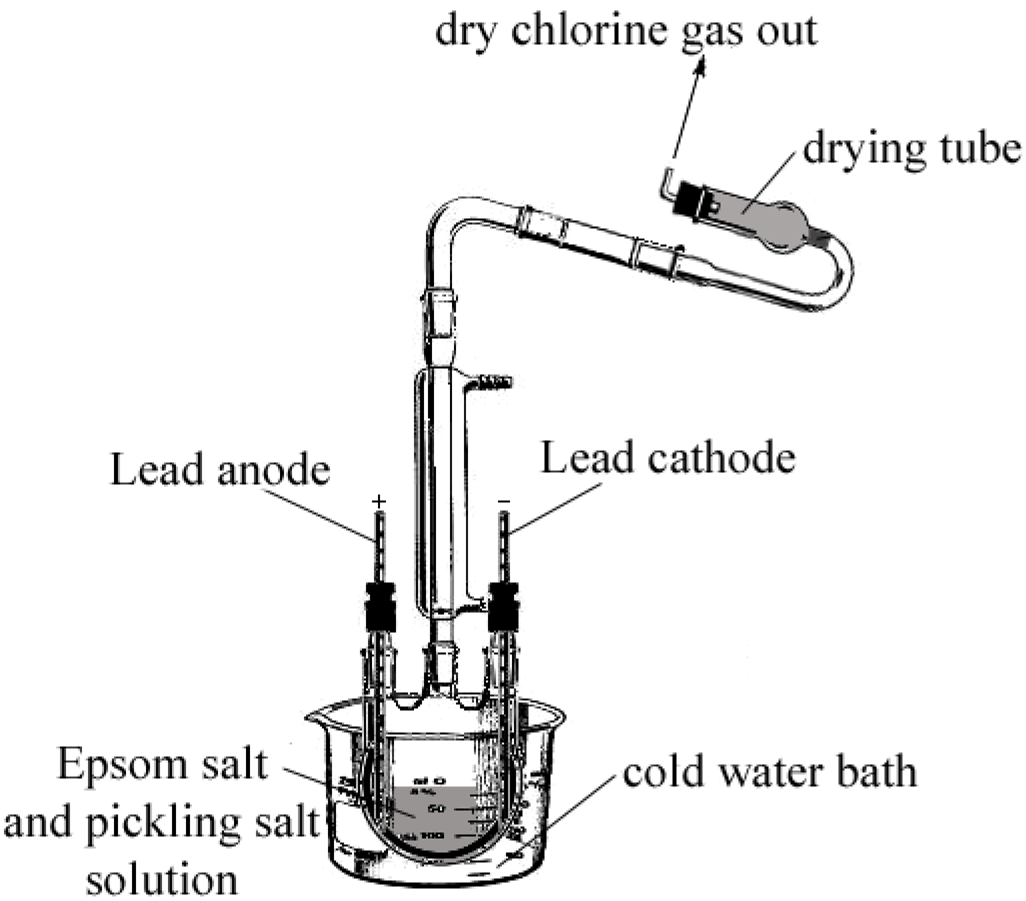
1. 200 grams (7 oz.) of Epsom salt	3. 95 grams (3.4 oz.) of pickling salt
2. 1000 milliliters (33.8 fluid oz.) of cold water	

Reaction summary: Chlorine gas can also be generated by electrolysis of a mixture of Epsom salt and pickling salt. The electrodes used should be made of lead to prevent excessive corrosion. During the reaction, water insoluble magnesium hydroxide will precipitate, and a steady stream of chlorine gas will be evolved over time. This procedure is useful in generating moderate amounts of chlorine gas while using simple and readily available materials.

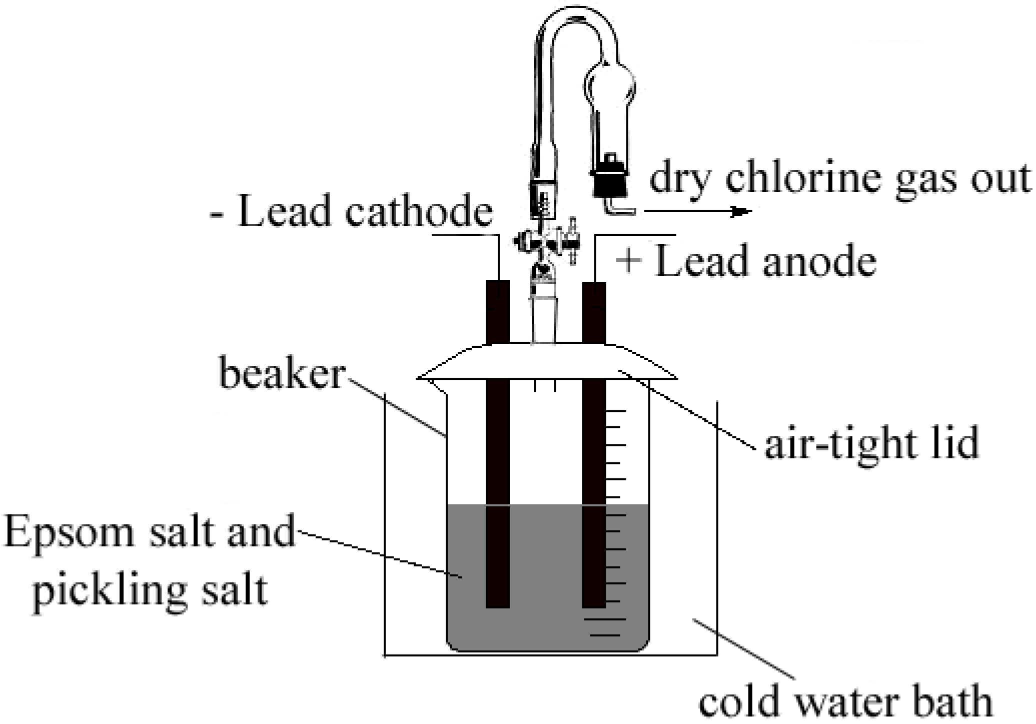


Hazards: Use proper ventilation when handing chlorine gas, and avoid contact with direct sunlight.

Procedure: Dissolve 200 grams (7 oz.) of Epsom salt into 1000 milliliters (3.8 fluid oz.) of cold water, and thereafter, add and dissolve 95 grams (3.4 oz.) of pickling salt. Now, pour this salt solution into an apparatus as illustrated below, and then electrolysis the mixture at 6 to 12 volt at 6 to 12 amp for a period ranging from 12 to 18 hours. During the electrolysis process, a moderate, yet steady stream of chlorine gas will be steadily evolved and carried over. NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that this electrolysis is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.



Setup for the generation of chlorine gas from readily available materials. As with the electrolysis of hydrochloric acid, keep the apparatus away from direct sunlight, magnesium light, or UV light.



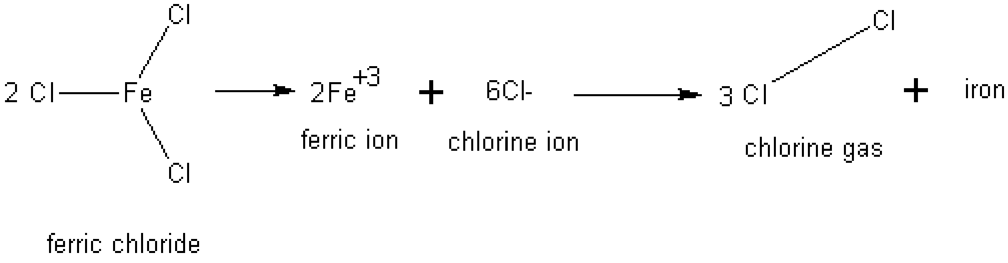
Setup for the preparation of chlorine. As with the electrolysis of hydrochloric acid, keep the apparatus away from direct sunlight, magnesium light, or UV light.

Preparation of hydrogen and oxygen free chlorine gas from ferric chloride utilizing an open cell

Materials:

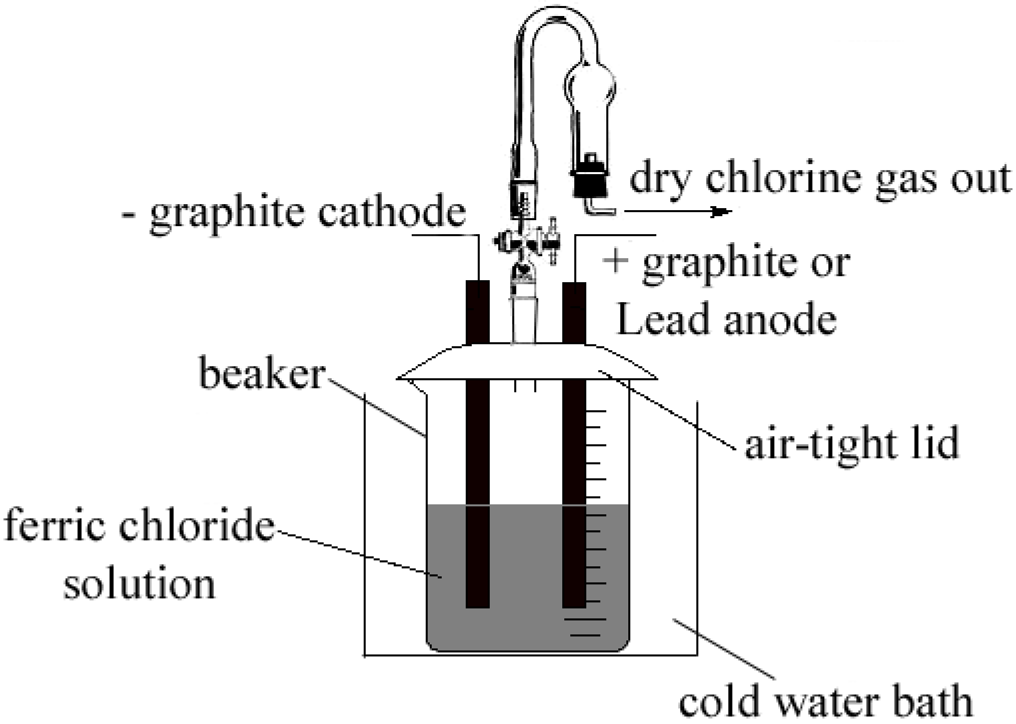
1. 400 milliliters (13.5 fluid oz.) of warm water	2. 200 grams (7 oz.) of ferric chloride hexahydrate

Reaction summary: **A oxygen and hydrogen gas free chlorine can be prepared by electrolyzing a solution of ferric chloride using an open cell. During the process, metallic iron will plate out on the cathode, and hence, can be collected for recycle.**



Hazards: Use proper ventilation when handing chlorine gas, and avoid contact with direct sunlight.

Procedure: Into an open cell as illustrated below, place 400 milliliters (13.5 fluid oz.) of warm water, and then add and dissolve 200 grams (7 oz.) of ferric chloride hexahydrate. Thereafter, electrolysis the solution from 6 to 12 volt, by 2 to 6 amp direct current for about 8 to 12 hours. During the electrolysis, a steady stream of hydrogen and oxygen free chlorine gas will be steadily evolved, and metallic iron will slowly plate the cathode. Note: The fresh metallic iron, can be recycled by using it as the anode for another electrolysis process using a diaphragm cell or divided cell, or it can be dissolved into hydrochloric acid directly if desired to prepare more ferrous chloride.



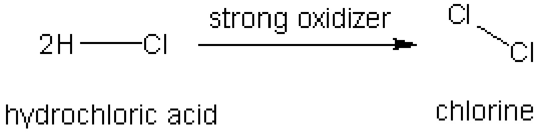
Setup for the preparation of hydrogen and oxygen free chlorine gas. During the electrolysis, metallic iron will slowly coat the iron cathode. Note: your apparatus can be sealed airtight by the use of plumber's putty, if your lid does not fit airtight, or there are gaps between the holes and your protruding electrodes.

Preparation of chlorine gas from hydrochloric acid and an oxidizing salt

Materials:

1. 75 grams (2.6 oz.) of manganese dioxide, or 1200 grams (2.6 pounds) of a 5% sodium hypochlorite solution (Clorox bleach), or 65 grams (2.3 oz.) of potassium permanganate, or 95 grams (3.3 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine)	3. 200 milliliters (6.7 fluid oz.) of tap water
2. 150 grams (5.3 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work)	

Reaction summary: Chlorine gas is prepared by dripping hydrochloric acid onto a strong oxidizing salt. During the reaction, chlorine gas is steadily evolved.

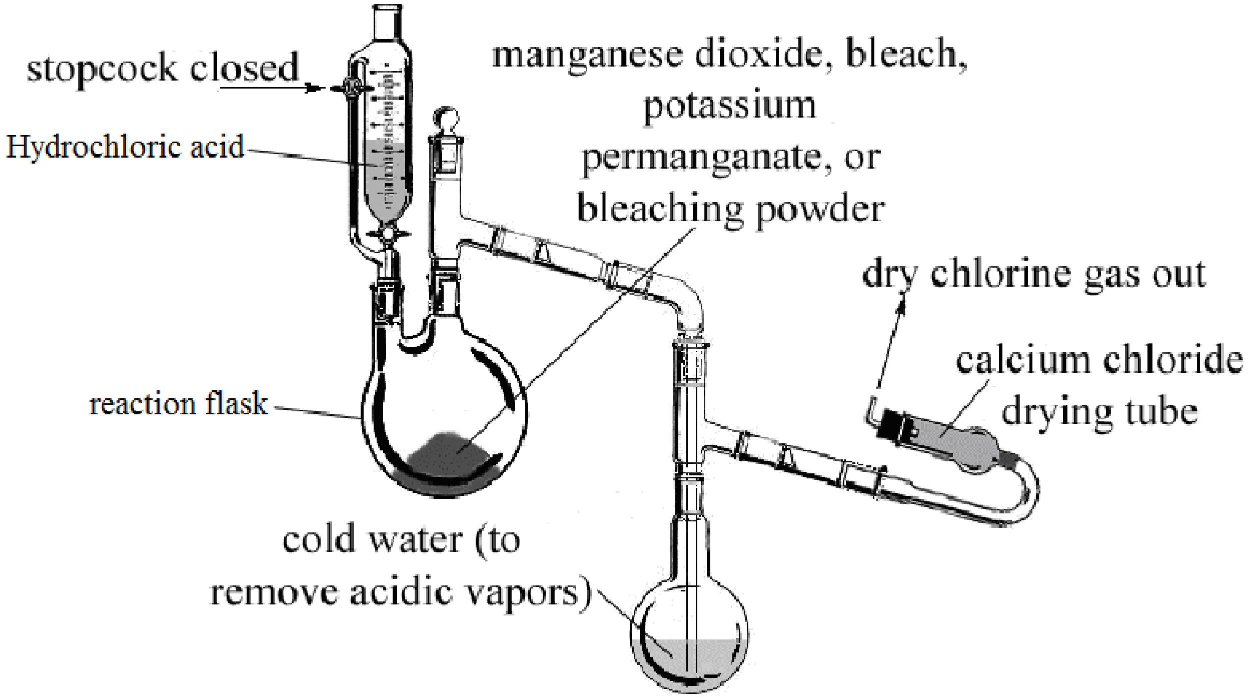


Hazards: Use care when handling strong oxidizers like manganese dioxide, potassium permanganate, sodium hypochlorite, and calcium hypochlorite, as they are all highly reactive and capable of reacting with many organic substances. Mixtures of strong oxidizers and combustible materials can ignite and burn violently. Wear gloves when handling hydrochloric acid, which can cause skin irritation. Chlorine is toxic, so handle with care and use caution.

Procedure: Setup the chlorine generating apparatus as illustrated below. Note: your chlorine generator does not have to be as fancy as the apparatus illustrated below, but your system should be similar in design. You can use latex tubing, PVC pipes and other plastic devices; as well, steel pipes can be used, but other metals should be avoided due to corrosion.

After you have the chlorine generator set-up, place into the reaction flask, 75 grams (2.6 oz.) of manganese dioxide, or 1200 grams (2.6 pounds) of a 5% sodium hypochlorite solution (Clorox bleach), or 65 grams (2.3 oz.) of potassium permanganate, or 95 grams (3.3 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 150 grams (5.3 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work) into 200 milliliters (6.7 fluid oz.) of tap water. Note: If using Clorox bleach, avoid brands that contain sodium hydroxide—look on the label to find out if your bleach has sodium hydroxide in it. Sodium hydroxide will screw-up the reaction, which is the main reason the companies add it in!

Once your chlorine generator is setup, begin slowly dripping the hydrochloric acid solution onto the oxidizer contained in the reaction flask. During the reaction, chlorine gas will be steadily evolved. Note: Obviously, you should have another apparatus set-up for reacting the chlorine with something.


stopcock closed
Hydrochloric acid
manganese dioxide, bleach, potassium permanganate, or bleaching powder
dry chlorine gas out
calcium chloride drying tube
reaction flask
cold water (to remove acidic vapors)

Setup for the generation of chlorine gas.

Chlorine dioxide

$$ClO_2$$

Chlorine dioxide forms a yellow to reddish yellow highly irritating gas, which is a strong oxidizer. The odor resembles that of chlorine and nitric acid. Chlorine dioxide is a unstable gas, that will detonate in contact with sunlight, mercury, and many organic materials. The gas can be safely used when diluted with an inert gas such as nitrogen or argon to form a 10% by weight mixture—however, even these gas mixtures can detonate in the presence of sunlight. Chlorine dioxide has a melting point of –59 Celsius, with a boiling point of 11 Celsius. Warning! Chlorine dioxide is a dangerous gas to work with, and when working with this gas a blast shield should be used in front of the apparatus in question. Chlorine dioxide is made by passing dry chlorine gas over finely powdered sodium chlorite at 50 Celsius under an inert atmosphere (excess inert gas) utilizing an all amber glass apparatus.

Copper, dust

Copper dust forms beautiful bright orangey-yellow particles that look like gold when thrown into a glass of water. The dust easily tarnishes in air, and will burn forming the oxide and volatizing forming copper vapors, which are toxic to the lungs. It can be made by electrolysizing a dilute sulfuric acid solution utilizing a copper anode, and a graphite cathode.

Copper turnings

$$Cu$$

Copper turnings form beautiful orangey metallic lustrous pieces. When heated, they oxidize in air forming copper-II-oxide. The turnings are made by dripping molten copper into distilled water containing sodium chloride brine, followed by filtration, and washing. Copper turnings are non-flammable.

Copper-II-chloride

$$CuCl_2$$

Copper-II-chloride, or cupric chloride forms yellow to brownish, deliquescent microcrystalline powder. The compound partially decomposes when heated to 300 Celsius forming chlorine gas and copper-I-chloride. Ordinary melting point is 630 Celsius. Cupric chloride forms a dihydrate when dissolved in water. The compound is soluble in water, alcohol, and acetone. It is made by first electrolysizing sodium chloride using copper electrodes in a divided clay pot cell, followed by recrystallization of the anode liquid, and then heating to 200 Celsius to remove the water of hydration.

Copper-I-oxide

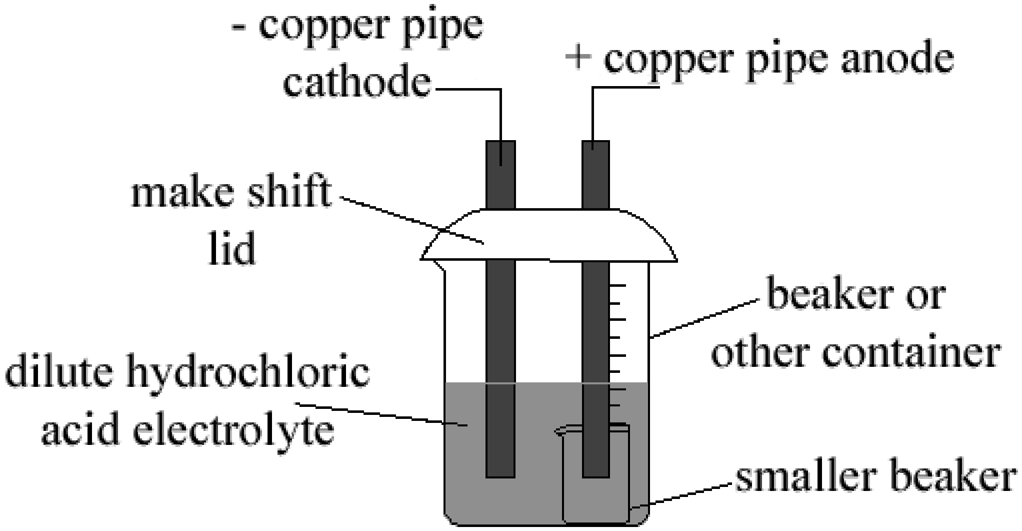
$$Cu_2O$$

Copper-I-oxide forms a brick red crystalline powder, which may be yellow, red, or brown depending on method of preparation. The powder is insoluble in water, and the usual solvents, but is readily soluble in acids, and ammonia solutions. The melting point of the powder is about 1232 Celsius, and the dry compound slowly oxidizes to copper-II-oxide on standing. Copper-I-oxide should be used within 1 month of its preparation.

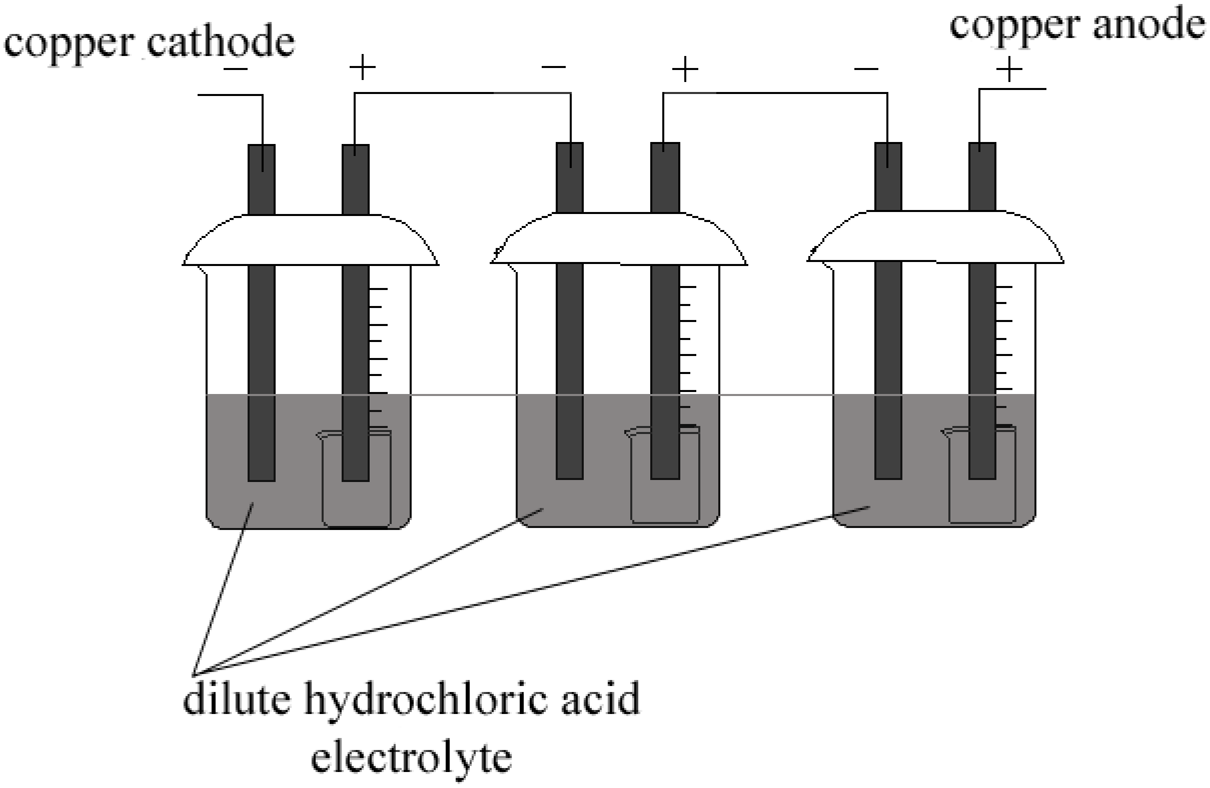
Preparation of copper-I-oxide

Procedure: set-up the apparatus illustrated below, and then add and dissolve 150 milliliters (5 fluid oz.) of concentrated hydrochloric acid or Muriatic acid into 750 milliliters (25.3 fluid oz.) of cold water, and then put in place two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding pipes, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. Note: A large volume of hydrogen gas will be steadily evolved from the electrodes so use proper ventilation and do not allow the gas to accumulate. The copper pipe anode will corrode and disappear at a steady rate. The electrolysis process will be quite fast, much faster then the previous two procedures, and the power supply will have to be turned-off once every 5 to 10 minutes, and the positive copper cathode will have to be quickly and temporally removed, and then scraped-clean using a spatula or any suitable utensil to recover a layer of copper-I-oxide that forms. After each scraping, simply scrape or place the brick red copper-I-oxide precipitate into any beaker or similar container, and store submerged under water temporarily, until all the desired amount of copper-I-oxide is collected. Note: Over time, the electrolyte will be consumed, as the hydrochloric acid is precipitated in the form of copper-I-chloride. To collect the copper-I-chloride precipitate, simply place a small beaker or similar container directly under the copper anode, submerged, so the white flaky precipitate of copper-I-chloride can be collected as it forms. As the electrochemical reaction proceeds, the acid in the electrolyte is consumed by the formation of the copper-I-chloride. Even though the acid gets consumed, the electrochemical reaction can continue as long as necessary by simply adding in a little more concentrated hydrochloric acid or Muriatic acid. The amount of acid added, should not exceed 150 milliliters so as to prevent your power supply form being damaged due to the high conductivity of the cell. However, to maximize power input, you can hook several cells in series to maximize electrical current consumption**.**

After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated brick-red copper-I-oxide (contained submerged under water in a beaker), and then wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the washed solids. Note: do not use heat to dry the copper-I-oxide as it is easily oxidized to copper-II-oxide. Now, for the copper-I-chloride, carefully extract the small submerged beaker (placed under the copper anode to collect the flaky precipitate of copper-I-chloride), using a suitable utensil or equivalent, and then carefully filter-off the white mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off copper-I-chloride with three 50-milliliter portions (three 1.6 fluid oz. portions) of cold alcohol, and then vacuum dry or air-dry the washed copper-I-chloride. Note: copper-I-chloride is rather unstable and tends to change colors when exposed to air and moisture.



Apparatus for the preparation of copper-I-oxide and copper-I-chloride. The copper-I-oxide forms on the cathode, and then copper-I-chloride collects in the small beaker as a white flaky precipitate. The lid should be non airtight, and is sole used to secure the copper pipes and keep them aligned in a good vertical position without falling over.



Apparatus for running more then 1 cell on the same power supply to maximum current efficiency. This "in series" type operation can be used for any electrolysis procedures discussed in this book, even if using a diaphragm cell, and is not restricted sole to copper-I-oxide and copper-I-chloride as in this procedure.

Copper-II-oxide

$$CuO$$

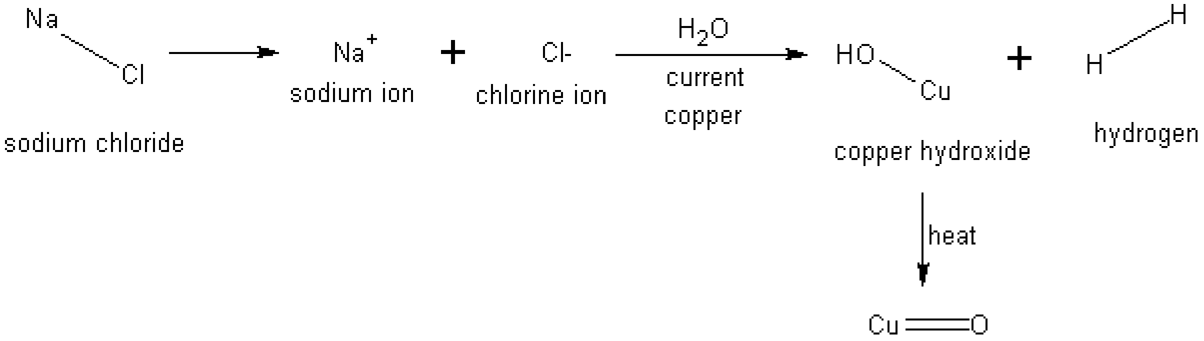
Copper-II-oxide forms a black to dark brownish-black amorphous or crystalline powder. The powder is insoluble in water and most organic solvents, but soluble in ammonia solution, acids and alkalis. The powder, after being roasted at high temperature for sometime, may become resistant to acids. Copper-II-oxide is widely used in the manufacture of fireworks and other pyrotechnic compositions for imparting a blue flame.

Method 1: Preparation of copper-II-oxide from copper pipes utilizing an open cell and pickling salt

Materials:

1. 100 grams (3.5 oz.) of pickling salt	3. Two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length
2. 400 milliliters (13.5 fluid oz.) of water	4. 750 milliliters of water (25.3 fluid oz.)

Reaction summary: Copper-II-oxide is formed in a similar manner as for iron-II-oxide. It is prepared, first, by electrolyzing a solution of pickling salt using copper electrodes. During the electrolysis process, a messy precipitate of mixed hydrated copper hydroxides are formed. Thereafter, this precipitate is collected by filtration, and then dried. The dry mass is then roasted at high temperature for several hours to facilitate formation of copper-II-oxide, which is formed by the dehydration and oxidization of the hydrated copper hydroxides.

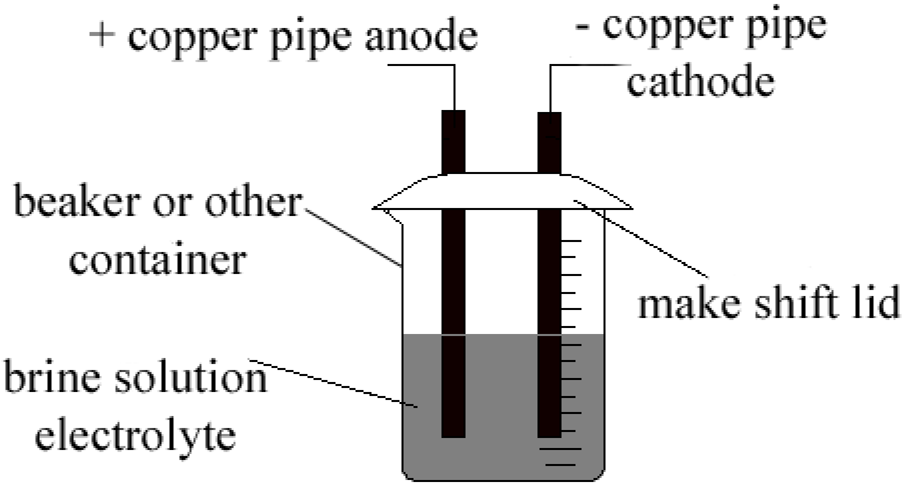


Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated copper hydroxides using an open electrochemical cell.

Set-up the apparatus illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding pipes, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a multi colored precipitate of mixed hydrated copper hydroxides will precipitate. The color will vary from yellowish to yellowish-orange. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. The copper pipe anode will slowly corrode and disappear. The electrolysis process can be slow, and can take any where from 12 to 16 hours to complete, depending on the thickness of the copper pipes or rods, and depending on how much of the copper oxide you want. After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated messy mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the filtered-off mass.

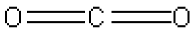


Set-up for the preparation of mixed hydrated copper hydroxides (practically identical to the iron oxide preparations).

Step 2: Preparation of copper-II-oxide

To prepare copper-II-oxide, all you need to do is place the dried mass of hydrated copper hydroxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatized and removed, and the copper hydroxides are oxidized to copper-II-oxide forming a black powder. After the roasting process, the copper-II-oxide is cooled, and then stored in any suitable container. This copper-II-oxide can be used in pyrotechnic compositions, or used as a catalyst for the oxidation of various gases, such as the oxidation of methanol to formaldehyde.

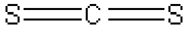
Carbon dioxide



Carbon dioxide

Carbon dioxide forms a colorless, odorless, non-toxic gas. At high concentrations carbon dioxide is a asphyxiate that displaces air and can cause systematic poisoning, but only when in high concentrations; other then that carbon dioxide is non-toxic, and is exhaled when you breathe. Carbon dioxide, also known as dry ice has a volatizing point of –78 Celsius. Carbon dioxide is produced upon combustion of fossil fuels, and is a major green house gas, which contributes to global warming and other weather changing effects. Carbon dioxide is easily made in the lab by treating baking soda with dilute acids. Carbon dioxide readily reacts with lithium or potassium hydroxide forming carbonates, and is hence, used in filters for spacecraft to remove traces of carbon dioxide from the air.

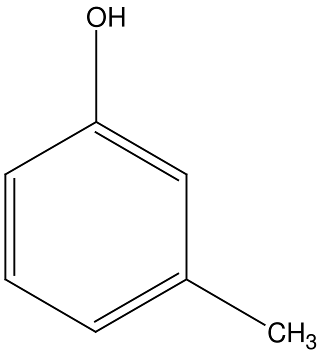
Carbon disulfide



Carbon disulfide

Carbon disulfide forms a highly refractive mobile flammable and poisonous liquid that has a boiling point of 46 Celsius, and a melting point of –111 Celsius. The liquid is readily flammable and burns with a bluish flame forming carbon dioxide and sulfur dioxide. Carbon disulfide is insoluble in water, but it has a tendency of forming an azeotrope containing 97% carbon disulfide and boiling at 42 Celsius. Carbon disulfide is miscible with alcohol, ether, benzene, chloroform, and fixed oils. Carbon disulfide is toxic so avoid skin contact, ingestion, and inhalation of the fumes. The liquid is made by roasting powdered charcoal with powdered sulfur at high temperature.

Meta-Cresol



meta-Cresol

meta-Cresol forms a colorless or yellowish , brownish-yellow, or pinkish liquid depending on purity with a phenolic odor. The liquid has a boiling point of 202 Celsius, with a melting point of 12 Celsius. The liquid is miscible with chloroform, alcohol, and ether.

Diborane gas

B_2H_6

Diborane gas forms a colorless flammable gas with an akward, repulsive odor with a melting point of –165 Celsius, and a boiling point of –92.5 Celsius. Decomposes at red heat forming boron and higher borane compounds. Diborane gas ignites spontaneously in air when heated to 40 to 50 Celsius; however contaminants may cause diborane gas to ignite spontaneous in air at room temperature. Mixtures of diborane gas with air can detonate under certain conditions so use caution. Diborane gas reacts with bromine and chlorine forming the boron halides. Diborane is made by direct union of powdered boron with hydrogen, followed by excessive fractional distillation to separate out diborane from the other boron hydrides.

Dinitrogen pentoxide

N_2O_5

Dinitrogen pentoxide forms colorless hexagonal crystals with a melting point of 30 Celsius. The crystals sublime when heated to 32.4 Celsius. The crystals are freely soluble in methylene chloride and chloroform without any excessive decomposition, but the solutions should be used within 24 hours of preparation. Even at room temperature the compound begins to decompose into oxygen and nitrogen oxides—can be stored for a week at –10 Celsius without excessive decomposition. Dinitrogen pentoxide is made by dehydrating nitric acid with phosphorus pentoxide in the presence of chloroform, followed by high vacuum distillation to remove the chloroform at low temperature until the single compound is left behind—the solid should then be recovered and stored in a freezer at –30 Celsius, but should be used within 1 week.

Distilled water

Distilled water is pure water with a boiling point of exactly 100 Celsius. 1 milliliter of water weights approximately 1 gram. It is made by distillation of tap water.

Dry ice

Dry ice forms a white snow like mass with a melting point of –79 Celsius. It is made by compressing carbon dioxide liquid/gas obtained from carbon dioxide tanks. Dry ice sublimes readily at room temperature skipping the liquid phase and transferring straight to a gas. Dry ice is readily available from numerous sources, and it can be stored in a freezer at –20 Celsius when sealed in Styrofoam boxes for up to a week.

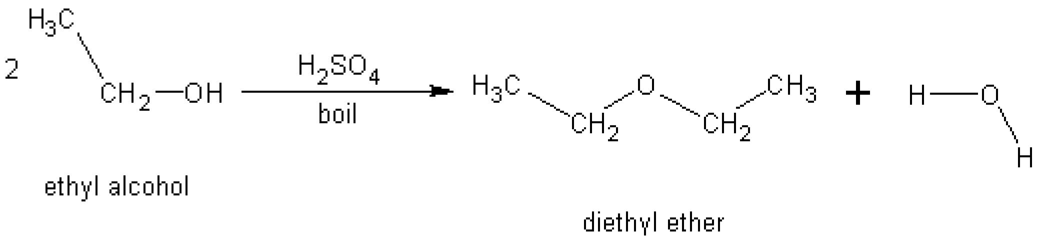
Diethyl Ether

Diethyl Ether, other wise known as just ether, is a mobile, very volatile, highly flammable liquid, which produces explosive vapors. It has a sweetish, pungent odor, and a burning taste. Ether forms explosive peroxides when exposed to air—ether containing peroxides will detonate if heated, shattering the glass vessel. Before heating mixtures containing ether, the peroxide test should be conducted. To test for peroxides, add five drops of ferrous chloride solution to the ether mixture. If a red or black color appears, peroxides are present. *Note: This test will not work properly if there are oxidizing agents in the ether mixture*. Ether can be stabilized by the addition of small amounts of naphthols, but this does not protect ether 100% from peroxide formation. Ether has a melting point of –116 Celsius, and a boiling point of 35 Celsius. Ether and air mixtures are explosive, so extinguish all flames and do not smoke when handling it. Protect ether from static electricity, which can also cause fire. Ether is insoluble in water, but miscible with alcohol, benzene, chloroform, and many oils. Do not mix 99% nitric acid with ether, as detonation will take place. Inhalation of ether vapors can produce intoxicating effects. These effects include feelings of euphoria, well-being, relaxation, and a general state of high. These effects can also lead to feelings of drunkness. Ether is a narcotic in high concentrations, but is not habit forming. Store ether in tightly sealed bottles in a cool place (preferably in a refrigerator). For prolonged storage, store ether over sodium sulfite and keep in a bottle filled to the top (to minimize the air space). Ether can be prepared by heating 95% ethanol and 98% sulfuric acid (1 to 1 ratio) to 100 Celsius, and simultaneously condensing the distilled-off vapors of ether. The ether is then purified by re-distillation. Ether is a widely available commercial chemical, but is status as an anesthetic may limit its sale to the general public.

Method 1: Preparation of diethyl ether from vodka and concentrated sulfuric acid

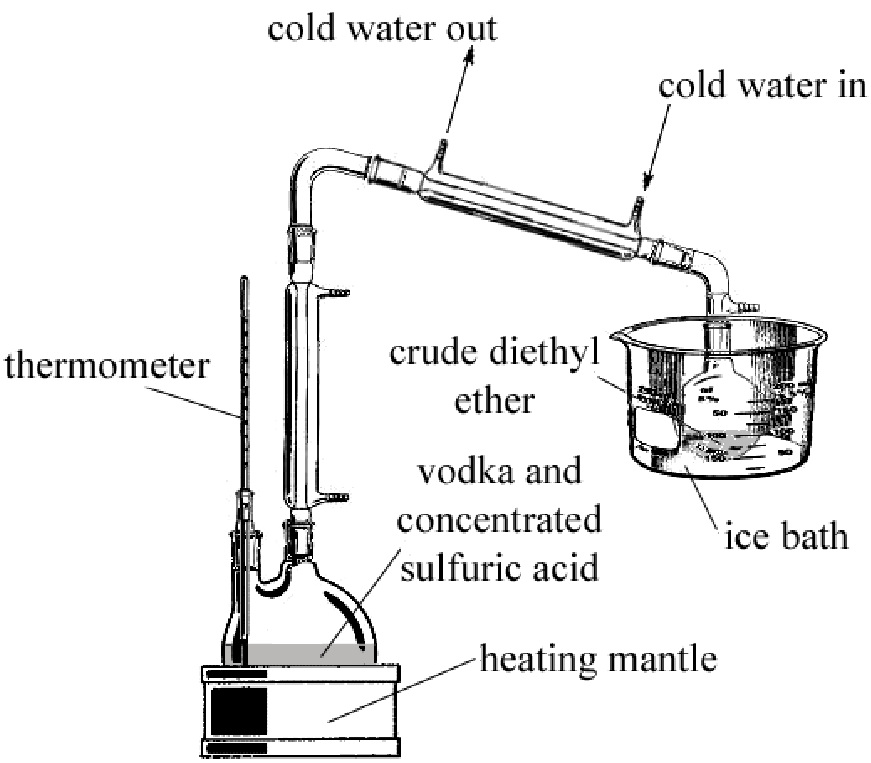
Materials:

1. 1250 milliliters (42.2 fluid oz.) of store bought vodka, usually 80 proof (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.,)	2. 900 milliliters (30.4 fluid oz.) of concentrated sulfuric acid (available at several places, but is readily available at janitorial and plumbing supply stores, where it sold as 93% to 98% purity. The 93% acid is 98% concentrated acid, but with iron, and can be used with satisfaction).



Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics.

Procedure: Into a suitable distillation apparatus, place 1250 milliliters (42.2 fluid oz.) of store bought vodka (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.,), followed by slowly and carefully adding in 900 milliliters (30.4 fluid oz.) of concentrated sulfuric acid. The concentrated sulfuric acid can be of any brand, and you may use plumbers brand concentrated sulfuric acid with satisfactory results. Note: The addition of the concentrated sulfuric acid to the vodka generates excessive heat, so use caution. After adding in all the concentrated sulfuric acid, boil the mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill over during this time. After 6 to 8 hours, remove the heat source, and allow the sulfuric acid mixture in the distillation flask or pot to cool to room temperature. The diethyl ether can then be recovered from the receiver flask, and then re-distilled using a fractional distillation apparatus, and distilled at 40 Celsius. The result after re-distillation will be around 500 milliliters of pure ether. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.



This apparatus can be replaced with any standard distillation apparatus, or standard fractional distillation apparatus; Alcohol stills can also be used, but may undergo corrosion in the distillation pot due to the presence of sulfuric acid. The diethyl ether should be re-distilled using a standard fractional distillation apparatus, or equivalent. After re-distillation, the ether should be stored in a proper container such as a metal can, or glass container, sealed air-tight, and filled to the top (to minimize air gaps), and these containers should be stored in a refrigerator until use. Note: the heating mantle portrayed in the illustration can be replaced by a Bunsen burner, hot plate, stovetop, steam bath, or oil bath if desired.

Method 2: Preparation of diethyl ether from vodka and battery acid

Materials:

1. 1250 milliliters (42.2 fluid oz.) of store bought vodka, usually 80 proof (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.,)	2. 1500 milliliters (50.7 fluid oz.) of fresh battery acid

Hazards: Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

Procedure: First, into a suitable sized thick walled Pyrex low form beaker, place 1500 milliliters (50.7 fluid oz.) of battery acid, and then boil the mixture by heating it to 150 Celsius until white fumes start to evolve. When white fumes start to evolve, turn off the heat source and allow the now concentrated sulfuric acid to cool to room temperature. Once it has, place it into a distillation apparatus as in method 1, and then add in all at once 1250 milliliters (42.2 fluid oz.) of vodka, and then distill the mixture at 100 Celsius for about 6 to 8 hours. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent and distill at 40 Celsius. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

Method 3: Preparation of diethyl ether from distilled ethyl alcohol (95% alcohol; ABS alcohol; ever clear), with concentrated sulfuric acid or battery acid

Materials:

1. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol; ABS alcohol; ever clear	3. or 500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear
2. 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid	4. 1500 milliliters (50.7 fluid oz.) of battery acid

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics. Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

Procedure: If using concentrated sulfuric acid, place 500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear), into a standard distillation apparatus, as illustrated in method 1 or similar, and then slowly and carefully add in 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid (plumbers grade works great). Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent in the same manner as in method 1 (distill at 40 Celsius). Note: the sulfuric acid mixture left over, can be recycled, but it should be boiled to drive-off some of the water.

If using battery acid, place into a suitable sized thick walled Pyrex low form beaker, 1500 milliliters (50.7 fluid oz.) of battery acid, and then boil the mixture by heating it to 150 Celsius until white fumes start to evolve. When white fumes start to evolve, turn off the heat source and allow the now concentrated sulfuric acid to cool to room temperature. Once it has, place it into a distillation apparatus as in method 1, and then add in slowly, 1500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear), as illustrated in method 1 or similar. Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent and distill at 40 Celsius. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

Method 4: Preparation of diethyl ether from denatured alcohol with concentrated sulfuric acid or battery acid

Materials:

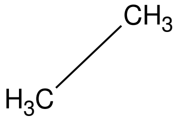
1. 550 milliliters (18.5 fluid oz.) of denatured alcohol—ethyl alcohol (available in most hardware stores, paint stores, and automotive stores).	3. or 550 milliliters (18.5 fluid oz.) of denatured alcohol
2. 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid	4. 1500 milliliters (50.7 fluid oz.) of battery acid

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics. Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

Procedure: If using concentrated sulfuric acid, place 550 milliliters (18.5 fluid oz.) of denatured alcohol into a standard distillation apparatus as illustrated in method 1 or similar, and then slowly and carefully add in 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid (plumbers grade works great). Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the crude diethyl ether from the receiver flask or receiver container. Finally, place this crude ether into a clean fractional distillation apparatus, and distill the ether at 40 Celsius, as in method 1. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

If using battery acid, place into a suitable sized thick walled Pyrex low form beaker, place 1500 milliliters (50.7 fluid oz.) of battery acid, and then boil the mixture by heating it to 150 Celsius until white fumes start to evolve. When white fumes start to evolve, turn off the heat source and allow the now concentrated sulfuric acid to cool to room temperature. Now, place 550 milliliters (18.5 fluid oz.) of denatured alcohol into a standard distillation apparatus, as illustrated in method 1 or similar, and then add in the concentrated battery acid previously prepared. Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the crude diethyl ether from the receiver flask or receiver container. Finally, place this collected crude ether layer into a clean fractional distillation apparatus, and distill the ether at 40 Celsius. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

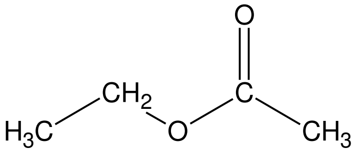
Ethane



Ethane

Ethane forms a colorless odorless highly flammable gas that comprises 9% of natural gas. The gas has a melting point of –172 Celsius, with a boiling point of –88 Celsius. Ethane is obtained during the destructive distillation of crude petroleum, and most of it is burned off on sight. Ethane gas is a narcotic in high concentrations.

Ethyl acetate



Ethyl acetate

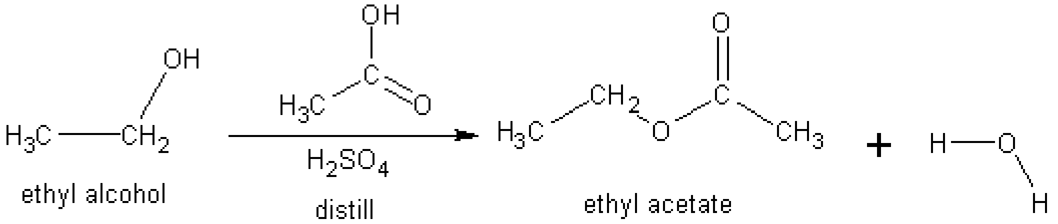
Ethyl acetate is a clear, volatile, and flammable liquid with a pleasant, fruity odor. It has a pleasant taste when diluted. Ethyl acetate slowly decomposes by moisture, so it should be kept in air-tight bottles and in a cool dry place. It has a boiling point of 77 Celsius, and a melting point of –83 Celsius. Ethyl acetate is moderately soluble in water (1 milliliter in 10 milliliters of water), but is miscible with alcohol, acetone, chloroform, and ether. It forms a azeotropic mixture with water (6% by weight with a boiling point of 70 Celsius). Ethyl acetate can be prepared by distilling a mixture of ethanol and acetic acid in the presence of a few drops of sulfuric acid. Ethyl acetate is a valuable solvent for many chemical reactions.

Method 1: Preparation of ethyl acetate from Vodka, battery acid, and vinegar

Materials:

1. 1000 milliliters (1 quart) of standard 5% acidity distilled crystal clear vinegar	4. 5 drops of concentrated sulfuric acid or 10 to 15 drops of battery acid
2. 180 milliliters (6 fluid oz.) of concentrated vinegar of 30% acidity	5. 15 grams (1/2 oz.) of anhydrous magnesium sulfate
3. 120 milliliters (4 fluid oz.) of cheap Vodka of 80 proof	

Reaction summary: Ethyl acetate is readily and easily prepared by distilling a mixture of vinegar and ethyl alcohol in the presence of a small amount of sulfuric acid. The sulfuric acid is the catalyst. The resulting distilled product is then treated with anhydrous magnesium sulfate and re-distilled to remove water.



Hazards: Use caution when handling sulfuric acid.

Procedure: Into a large distillation apparatus, or similar distillation apparatus of any desired type or design, place 1000 milliliters (1 quart) of standard 5% acidity distilled crystal clear vinegar or 180 milliliters (6 fluid oz.) of concentrated vinegar of 30% acidity, followed by 120 milliliters (4 fluid oz.) of cheap Vodka of 80 proof, and then followed by 5 drops of concentrated sulfuric acid, or 10 to 15 drops of battery acid. Thereafter, heat and distill the mixture at 77 Celsius until no more ethyl acetate distills over—the time may vary from 1 to 3 hours. After the distillation period, the crude ethyl acetate should be thoroughly mixed with about 15 grams (1/2 oz.) of anhydrous magnesium sulfate, and stirred for about 30 minutes at room temperature. Finally, filter-off the magnesium sulfate, and then fractionally distill the filtered liquid at 77 Celsius to obtain a refined product of at least 99% ethyl acetate.

95% Ethyl alcohol

95% Ethanol is a clear, colorless, very mobile, flammable liquid with a pleasant odor, and a pungent, burning taste. It has a boiling point of 78 Celsius and a melting point of –114 Celsius. 95% Ethanol slowly absorbs water from the air, and dilute ethanol solutions are slowly oxidized by air forming brown colored solutions containing mixtures of aldehydes, and carboxylic acids; mainly acetic acid. 95% Ethanol is miscible with water, and many organic solvents. 95% ethyl alcohol is called absolute ethanol because ethyl alcohol forms a binary azeotrope containing 95.57% ethyl alcohol by weight with a boiling point of 78 Celsius. Distillations cannot produce 99% ethanol because of this azeotrope. Ethyl alcohol is usually sold as denatured ethyl alcohol (mixed with small amounts of toxic chemicals to make non-drinkable) due to US government tax regulations. 95% Ethyl alcohol is toxic, and ingestion can cause alcohol poisoning. Dilute mixtures of ethyl alcohol (Vodka, Gin, Rum, Jack Daniels, beer, wine) produce intoxicating effects when ingested (these intoxicating effects can be increased if the dilute ethanol mixture is injected). 95% ethanol can be made by fermenting starch or sugars with yeast, followed by double distillation. 95% Ethanol is manufactured on an industrial scale by the petroleum industry from ethylene gas, sulfuric acid, and water. 95% Ethanol is a widely available commercial chemical sold under a variety of names. 95% ethanol can be obtained from double distillation of alcoholic beverages such as vodka, gin, or rum.

99% Ethyl alcohol

99% Ethyl alcohol is a colorless, very mobile and flammable liquid with a pleasant odor. Pure ethanol is tasteless. It rapidly absorbs water from the air, from which is forms an azeotrope of 95% ethyl alcohol. It is miscible with water, alcohol, ether, and many common organic solvents. 99% ethanol is toxic, and ingestion can cause poisoning. It is prepared by reacting ethylene gas with sulfuric acid, followed by distillation in the presence of minute amounts of water. It can also be made by double distillation of fermented cocktails, followed by treatment with metallic sodium to remove the water of azeotrope. 99% Ethyl alcohol can be obtained by salting out vodka (see 91% isopropyl alcohol), treating the recovered upper ethyl alcohol layer with large amounts of anhydrous magnesium sulfate, filtering, and then distilling the mixture to recover 99% ethyl alcohol.

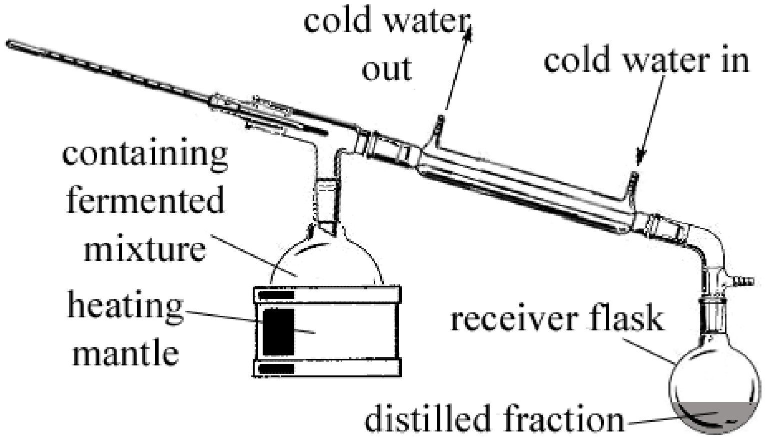
Method 1: Preparation of 95% ethyl alcohol from liquor, wine, or beer

Materials:

1. 1 liter or more (1.86 quarts or more) of 60 to 180 proof liquors (any kind will work, but you should use the cheap stuff to save money)	3. or 5 liters or more (1.3 gallons or more) of any beer, ail, brew, or malt liquor
2. or 2.5 liters or more (2.64 quarts or more) of wine (any will work) with an alcohol concentration of at least 7%	4. 50 grams (1.7 oz.) of baking soda

Procedure: If using liquors, place 1 liter or more (1.86 quarts or more) of 60 to 180 proof liquors (any kind will work, but you should use the cheap stuff to save money), into a clean distillation apparatus or similar as illustrated below. If using wines, place 2.5 liters or more (2.64 quarts or more) of wine (any will work) with an alcohol concentration of at least 7%, into a distillation apparatus (similar to the one illustrated below). If using beer, place, 5 liters or more (1.3 gallons or more) of any beer, ail, brew, or malt liquor into a distillation apparatus or similar device as illustrated below. Then, despite which alcoholic beverage you use, distill your alcoholic beverage or concoction at 90 Celsius until no more liquid passes into the receiver flask (will take 4 to5 hours for the liquors, 4 to 8 hours for the wines, and 6 to 10 hours for the beers). When no more liquid passes into the receiver flask, remove the heat source, and then remove the receiver flask from the distillation apparatus. Then place 50 grams (1.7 oz.) of baking soda into the receiver flask, and then boil the contents to 100 Celsius for about 5 minutes. Thereafter, remove the heat source, and allow the contents to cool to room temperature. Afterwards, filter the mixture to remove the insoluble baking soda, and then place this filtered mixture into a clean fractional distillation apparatus. Then distill at 80 Celsius until no more liquid passes into the receiver flask.

First distillation



Simple distillation apparatus for the distillation of liquors, wines, or beers to yield 90%+ ethyl alcohol.

Method 2A: Preparation of 95% ethyl alcohol from table sugar or common food grade starch (corn starch)

Materials:

1. 1 kilogram (2.2 pounds) of table sugar	7. or 2 liters (2.1 quarts) of hot tap water
2. 3 liters (3.2 quarts) of tap water	8. or 10 drops of concentrated hydrochloric acid (muriatic acid will work).
3. 5 drops of concentrated hydrochloric acid (muriatic acid will work) or 5 drops of concentrated sulfuric acid (plumbers grade will work).	9. or 1 small spoon full of baking soda
4. 115 grams (4 oz.) of baking soda	10. or 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies)
5. 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies)	11. 50 grams (1.8 oz.) of baking soda
6. or 500 grams (17.6 oz.) of regular store bought food starch not the "starch" used for clothing	12. Charcoal (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.,

Procedure:

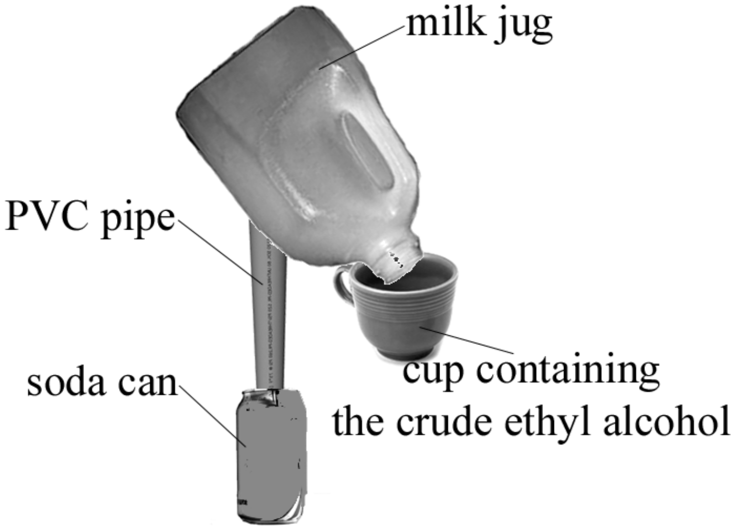
If using table sugar:

Dissolve 1 kilogram (2.2 pounds) of table sugar into 3 liters (3.2 quarts) of tap water. Then rapidly stir this sugar mixture to dissolve all solids, and thereafter, heat this sugar solution (using a hot plate, stovetop, Bunsen burner, ect.,) to 80 Celsius. When the sugar solution reaches about 80 Celsius, stir the sugar solution and add in 5 drops of concentrated hydrochloric acid or 5 drops of concentrated sulfuric acid (hydrochloric acid works best), and then continue heating and stirring the sugar solution for thirty minutes (at the end of 15 to 30 minutes, the sugar solution should have a slight grape sugar like odor). After thirty minutes, remove the heat source, and allow the sugar mixture to cool to room temperature. Then add in 5 grams (2/10th of an oz.) of baking soda to neutralize the acid. Afterwards, pour the sugar solution into an empty bottle (such as a clean empty plastic milk jug), and then add in 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies). Note: the yeast must be finely ground into a small amount of warm water before adding to the sugar solution. After adding the yeast slurry, stir the mixture for several minutes to insure good dispersion of the yeast. Then plug the bottles opening with cotton or similar materials (but not air-tight), and then place the bottle into a cool place away from light. Then allow the sugar mixture to ferment for about 2 to 3 weeks. After about 2 to 3 weeks, remove the cotton or similar material from the bottles opening, and then pour the contents of the bottle into a distillation apparatus (similar to the one illustrated in method 1). Then distill at 100 Celsius for 3 to 4 ½ hours. After which, remove the heat source, and then recover the crude ethyl alcohol mixture from the receiver flask. Then pour this crude ethyl alcohol mixture into a clean beaker or other container, and then add in 50 grams (1.8 oz.) of baking soda. Thereafter, boil this mixture for about 5 minutes, and thereafter, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, filter the mixture to remove any insoluble baking soda, and then place the filtered mixture into a regular clean distillation apparatus, and distill at 80 Celsius until no more liquid passes into the receiver flask (takes about 3 to 4 hours). When no more liquid passes into the receiver flask, remove the heat source, and then recover the crude ethyl alcohol from the receiver flask just like before. Finally, place this crude ethyl alcohol mixture into a clean fractional distillation apparatus followed by 50 grams (1.8 oz.) of baking soda, and then distill the mixture at 80 Celsius until no more ethyl alcohol passes into the receiver flask (takes about 3 to 4 hours). Once the ethyl alcohol has distilled over for this final time, recover the ethyl alcohol from the receiver flask, and then filter it through a filter (gravity filtration) containing a layer of charcoal. Note: simply place ordinary charcoal briquettes (crush them up into pieces if necessary) into the bottom of the filter paper (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.,). Pass the ethyl alcohol through this filter several times to purify it—the charcoal will help remove impurities, odors, and the like. After the charcoal filtration process, your 95% ethyl alcohol is ready for use. Note: if desired, this alcohol can be used to prepare ether, or diluted to 100 to 80 proof for consumption!

If using starch

Into a large beaker or other container, place 500 grams (17.6 oz.) of regular store bought food starch (not the "starch" used for clothing). Thereafter, add in 2 liters (2.1 quarts) of hot tap water, and then add in 10 drops of concentrated hydrochloric acid. Thereafter, heat this starch suspension for about 2 hours at the boiling temperature of water. During the heating process, stir the mixture as much as possible. Keep heating the mixture until the bulk of the starch dissolves. Note: The purpose for the acid is to hydrolyze the starch into glucose. During the heating process, the insoluble starch suspension will slowly disappear to give a clear solution; this is because of the starch being broken down into glucose, which is very soluble in water. When most of the insoluble starch has been dissolved, remove the heat source, and allow the resulting solution to cool to room temperature. When the solution cools, add in 1 small spoon full of baking soda (to neutralize the acid), and then pour the entire mixture into a clean empty plastic milk jug. At his point, you should now have a plastic milk jug filled with just over 2 liters of glucose solution, containing a small amount of baking soda. Now, pour in 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies). Note: the yeast needs to be finely ground in a small amount of warm water to form a slurry. Thereafter, when the yeast has been added, don’t shake the container; just allow the yeast to settle on its own. After adding in the yeast, plug the top of the plastic milk container with a cloth, cotton, or any thing that is not airtight, but will keep dust out. Thereafter, place your milk jug into a cool dark place, and let it ferment for about 2 to 3 weeks. After 2 to 3 weeks, your home brew is finished. In some cases, it may take as long as 4 weeks to complete the fermentation. Note: During the fermentation process, the yeast catalytically reacts with the glucose forming ethyl alcohol, and carbon dioxide. The carbon dioxide bubbles to the surface, and the alcohol remains in solution. Once the fermentation process is complete, it's now time for the hard part—the mixture has to be distilled. To do this, assemble an apparatus similar to the one in method 1, and then distill at 100 Celsius for 4 ½ hours. After which, remove the heat source, and then recover the crude ethyl alcohol mixture from the receiver flask. Then pour this crude ethyl alcohol mixture into a clean beaker or other container, and then add in 50 grams (1.8 oz.) of baking soda. Thereafter, boil this mixture for about 5 minutes, and thereafter, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, filter the mixture to remove any insoluble baking soda, and then place the filtered mixture into a clean fractional distillation apparatus, and distill at 80 Celsius until no more liquid passes into the receiver flask (takes about 3 to 4 hours). When no more liquid passes into the receiver flask, remove the heat source, and then recover the ethyl alcohol from the receiver flask. Then, filter it through a filter (gravity filtration) containing a layer of charcoal. Note: simply place ordinary charcoal briquettes (crush them up into pieces if necessary) into the bottom of the filter paper (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.,). Pass the ethyl alcohol through this filter several times to purify it—the charcoal will help remove impurities, odors, and the like. After the charcoal filtration process, your 95% ethyl alcohol is ready for use. Note: if desired, this alcohol can be used to prepare ether, or diluted to 100 or 80 proof for consumption!

NOTE: If you don't have access to laboratory glassware (which most people don’t), you will need to use your imagination and come up with a homemade "still" of your own. Use your creativity to design a cheap home made "still". One method is screwing a PVC pipe of appropriate size into the opening of a clean soda can, and then inserting the other end of the PVC pipe into an empty plastic container (such as an empty milk jug). Note: Do not drink the distilled alcohol fraction as is; its 90% alcohol and ingestion can cause poisoning, and it should be diluted before consumption.



Crude setup for the distillation of ethyl alcohol. In the above illustration, the fermented mixture is placed into the soda can and a PVC pipe is then inserted into the mouth opening. Plumbers putty or hot glue can be used to seal the PVC pipe to the soda can. A milk jug is then attached to the top of the PVC pipe, Plumbers putty or hot glue can be used to secure the PVC pipe to the milk jug. As heat is applied to the soda can, the ethyl alcohol will boil-off, and its vapors will condense on the roof of the milk jug. The condensate will then drip down into the cup, where the alcohol fraction can be obtained. Instead of using a fermented mixture, cooking wine can be used as well, but should be diluted before consumption.

Method 2B: Preparation of 95% ethyl alcohol from table sugar, brown sugar, molasses, or juice (improved process)

Materials:

1. 1 kilogram of sugar (2.2 pounds) or 1 kilogram (2.2 pounds) of brown sugar	5. 2 to 4 liters (2.1 to 4.2 quart) container of grape juice, apple juice, or apricot juice
2. 3 liters (3.1 quarts) of warm tap water	6. 1 kilogram (2.2 pounds) of molasses
3. 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies)	7. 3 liters (3.1 quarts) of warm water
4. 200 grams (7 oz.) of pickling salt	

Hazards: None.

Procedure: 95% Ethyl alcohol can be prepared by fermenting sugar, brown sugar, molasses, or juice using yeast in a similar manner to method 2A. However, in the case of sugar, instead of hydrolyzing the sugar, it is fermented directly using brewers yeast used for wine making. Now, if using sugar or brown sugar, dissolve 1 kilogram of sugar (2.2 pounds) or 1 kilogram (2.2 pounds) of brown sugar into 3 liters (3.1 quarts) of warm tap water contained in a washed out empty milk gallon jug, and then prepare a yeast base mixture by doing the following: 1. take 1 packet brewers yeast—7 grams or 1/4th of an ounce, and place it into 1/4th of a cup of warm water, and then using a spoon or similar utensil, grind and thoroughly mix the yeast into the water until a uniform mixture results, which is free from clumps, chunks, or the like. Thereafter, pour the yeast mixture into the sugar solution, and then place a paper towel or cloth over the mouth of the gallon jug, and then allow the mixture to ferment for about 2 weeks at room temperature. Note: once the yeast gets going, in about an hour or so the evolution of carbon dioxide will become vigorous. The total amount of time for the fermentation process ranges from about 14 to 16 days. When the evolution of carbon dioxide gas bubbles slows down and drops-off to a trickle, the fermentation process is relatively complete. After the fermentation process, the mixture needs to be distilled using a similar apparatus as in method 1. The distillation process will take about 5 to 6 hours for completion. After the distillation process, the crude alcohol mixture should then be "Salted out" using the following process: 1. Place the crude alcohol mixture into a separatory funnel (see extraction), and then add in 200 grams (7 oz.) of pickling salt, and then shake the mixture vigorously for about 5 to 10 minutes. Thereafter, allow the mixture to settle and stand for about 15 minutes, whereupon a two-phase mixture will develop. The upper layer will be the alcohol layer, and the water layer containing dissolved pickling salt and other impurities, will be the lower layer. Now, simply drain-off the lower water layer, and discard, or you can keep it to recover the dissolved salt, and then drain-off or pour out the upper alcohol layer. Then place this alcohol layer into a clean distillation apparatus of the usual design, as illustrated in method 1, and then distill the alcohol at 78 Celsius. The resulting alcohol may not be exactly 95%, but is well suitable for use in applications as a solvent.

If using molasses, or juice, simply prepare a yeast mixture by taking 1 packet of brewers yeast—7 grams or 1/4th of an ounce, and place it into 1/4th of a cup of warm water, and then using a spoon or similar utensil, grind and thoroughly mix the yeast into the water until a uniform mixture results, which is free from clumps, chunks, or the like, and then pour this yeast mixture directly into a large (2 to 4 liter, 2.1 to 4.2 quart) container of grape juice, apple juice, apricot juice, or a molasses solution, and then allow the mixture to ferment for about 2 weeks. As in the above process, once the yeast gets going, in about an hour or so the evolution of carbon dioxide will become vigorous. The total amount of time for the fermentation process ranges from about 14 to 17 days. When the evolution of carbon dioxide gas bubbles slows down and drops-off to a trickle, the fermentation process is relatively complete. Note: if using juice, pour out some of juice before adding the yeast to create a gap or space between the juice and the mouth of the container to prevent foaming and frothing from escaping the bottle once the yeast kicks in. Second not: If using molasses, dissolve 1 large container of molasses (about 1 kilogram, 2.2 pounds), into 3 liters (3.1 quarts) of warm water contained in a clean empty milk jug or equivalent. If using molasses, you will want to carryout the procedure in a well-ventilated area as the odor of molasses will be heavy. After the fermentation process, the fermented juice or molasses mixture needs to be distilled using a similar apparatus as in method 1. Then, place the crude ethyl alcohol mixture into a clean fractional distillation apparatus and then distill at 80 Celsius. The distillation process will take about 5 to 6 hours for completion. After the distillation process, the purified alcohol will be exactly 95%, and is well suitable for use in applications as a solvent, making ether, or for consumption (dilute to 100 or 80 proof for consumption!).

Method 3: Preparation of 99% ethyl alcohol from 95% ethyl alcohol and metallic sodium

Materials:

1. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol	2. 40 grams (1.4 oz.) of metallic sodium

Hazards: Wear gloves when handling metallic sodium, and avoid skin contact and ingestion. Metallic sodium reacts violently with water and other chemicals, so use caution.

Procedure: Into a suitable beaker or container, place 500 milliliters (17 fluid oz.) of 95% ethyl alcohol, and then place the beaker or container into an ice bath, and chill to about 5 to 10 Celsius. Thereafter, slowly add in, in very small portions, 40 grams (1.4 oz.) of metallic sodium over a period of about 1 hour. After the addition of the metallic sodium, stir the entire alcohol mixture for about 30 minutes, and then place it into a standard distillation apparatus, and distill the ethyl alcohol at 80 Celsius. Note: your distillation apparatus should be completely dry before proceeding. When no more ethyl alcohol passes into the receiver flask, remove the heat source, and then recover the ethyl alcohol from the receiver flask. The resulting alcohol will be about 99% pure, and should be stored in a suitable container protected from moisture. Note: never consume 99% ethyl alcohol as death may occur. 99% ethyl alcohol is never used to make alcoholic beverages and is primarily used as a solvent in organic reactions. Note: 99% ethyl alcohol is a controlled substance according to the US government.

Ferric Chloride, Anhydrous

$$FeCl_3$$

Ferric chloride forms hexagonal, dark leaflets or plates. The crystals have a melting point of 300 Celsius, with sublimation taking place. The crystals are readily soluble in water forming the hexahydrate, and soluble in alcohol, ether, acetone, with slight solubility in carbon disulfide. The anhydrous salt is formed by passing dry chlorine over small pieces of cast iron.

Ferric chloride hexahydrate

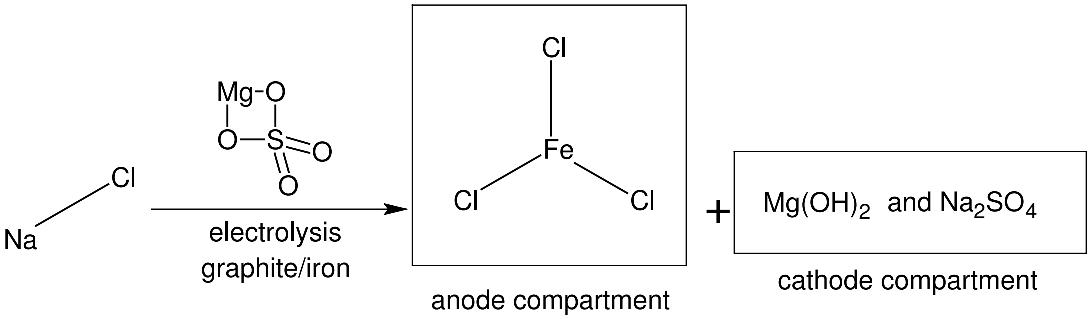
$$FeCl_3\text{-}7H_2O$$

Forms brownish yellow or orange monoclinic crystals, with a slight odor of hydrogen chloride. The melting point of the crystals is around 37 Celsius. The compound is readily soluble in water, alcohol, acetone, and ether.

Preparation of ferric chloride hexahydrate from pickling salt, Epsom salt, and iron/graphite electrodes

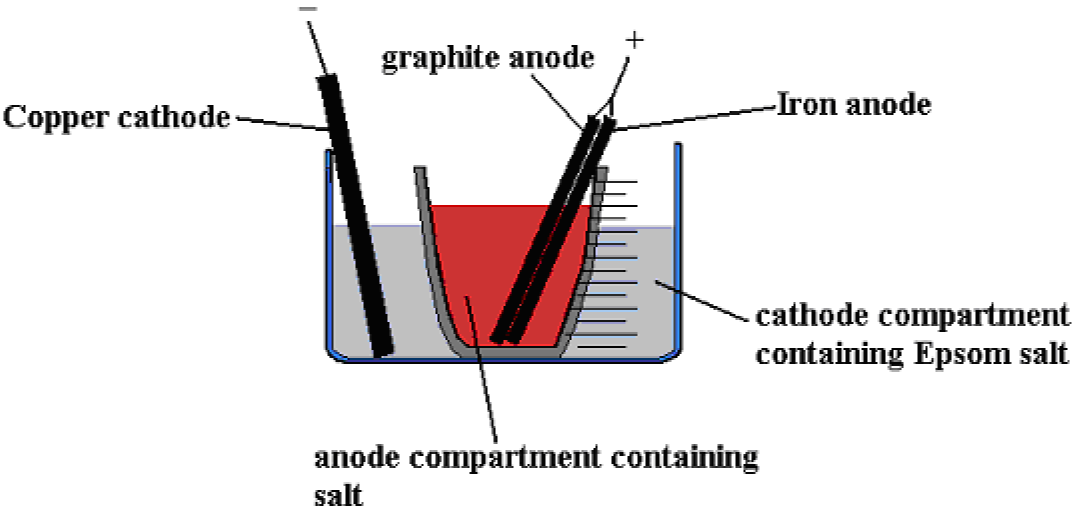
Materials:

1. 120 grams (4.2 oz.) of sodium chloride	3. 251 grams (8.8 oz.) of Epsom salt
2. 1100 milliliters (37 fluid oz.) of tap water	



Hazards: Use proper ventilation as hydrogen gas is evolved and accumulate in enclosed areas forming explosive mixtures.

Procedure: Dissolve 120 grams of sodium chloride into 400 milliliters of tap water. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 251 grams of Epsom salt into 700 milliliters of tap water, and then pour this solution into the cathode compartment. Then electrolysize the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/graphite anode, and copper cathode. After about 12 hours, remove the electrical source, and then filter the anode liquid to remove any insoluble materials. Then, place the filtered anode liquid into a crystallizing dish and allow it to evaporate to form brilliant golden yellow crystals. The cathode liquid can also be filtered to recover the insoluble magnesium hydroxide. If desired, the filtered cathode liquid can be evaporated to recover colorless crystals of sodium sulfate decahydrate.


−
+
graphite anode
Copper cathode
Iron anode
cathode compartment containing Epsom salt
anode compartment containing salt

Apparatus for producing ferric chloride in the anode compartment.

30% Ferric chloride hexahydrate solution

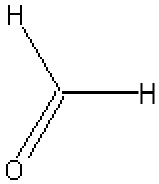
Prepare by dissolving 100 grams of ferric chloride hexahydrate into 230 milliliters of water.

Fluorine

$$F_2$$

Fluorine forms a pale yellow diatomic gas with a melting point of –219 Celsius, and a boiling point of –188 Celsius. The gas is highly toxic so inhalation should be avoided at all cost. Fluorine is the most reactive element known to man, and it reacts with many organic materials, often causing fires. Fluorine also reacts with water forming a mixture of products, and reacts with other inorganic compounds such as nitric and sulfuric acids forming explosive gases. Fluorine is prepared by electrolysizing a solution of potassium fluoride dissolved in liquid hydrogen fluoride at –23 Celsius using platinum electrodes.

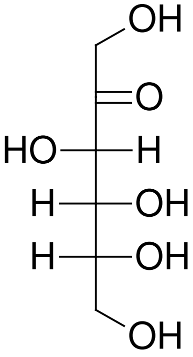
Formaldehyde



Formaldehyde

Pure formaldehyde is a colorless flammable gas with a pungent suffocating odor. The pure gas has a melting point of –92 Celsius, and a boiling point of –19 Celsius. In most cases, formaldehyde is handled as a 37% solution in water, from which methanol has been added to prevent polymerization. 37% formaldehyde solution is a colorless liquid with a pungent odor. Vapors from 37% formaldehyde can produce dizziness and headache upon inhalation, and maximum ventilation should be used when using. The colorless liquid solution may become cloudy on standing as a result of polymerization. 37% Formaldehyde slowly oxidizes to formic acid on standing. The liquid solution has a boiling point of 96 Celsius. It is miscible water, alcohol, and acetone

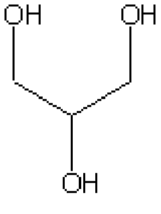
D-Fructose



D-fructose

D-Fructose forms orthorhombic, bisphenoidal prisms from alcohol. Begins to decompose when heated to 105 Celsius. The compound is sweater then table sugar. It is freely soluble in water, and only moderately soluble in alcohol, and insoluble in most organic solvents. It is prepared by enzymatic break down of table sugar. It can also be obtained by heating invert sugar obtained by acid hydrolysis of table sugar.

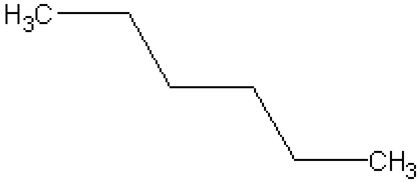
Glycerol. *1,2,3-Propanetriol; Glycerin*



Glycerol

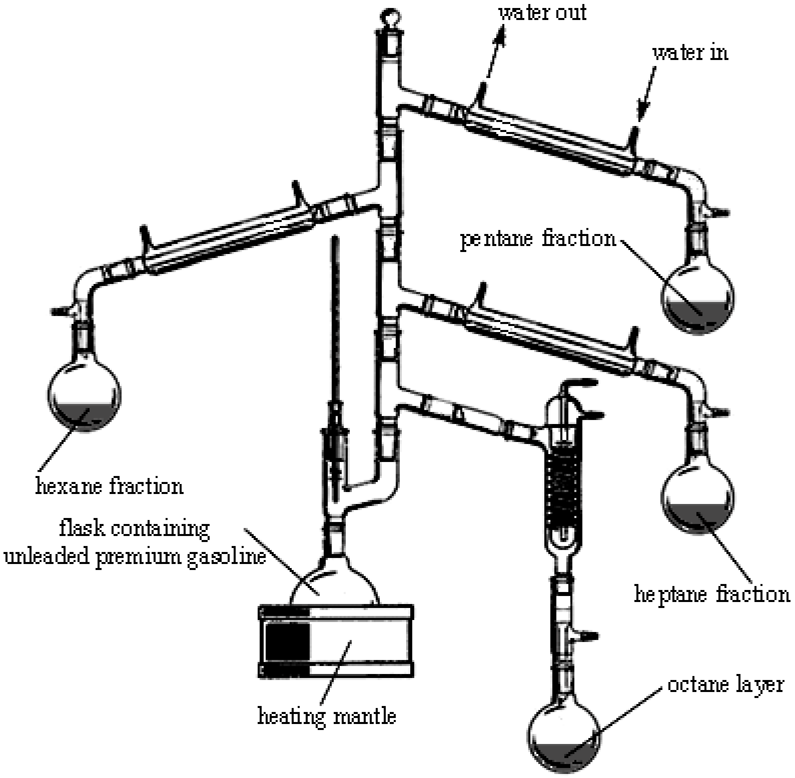
Glycerol is a syrupy liquid with a sweet warm taste. Glycerol is less sweet then cane sugar. It absorbs moisture from the air, and also absorbs gases like hydrogen sulfide, hydrogen cyanide, and sulfur dioxide. Explosions may result if glycerol is treated with strong oxidizers such as chromium trioxide, potassium chlorate, or potassium permanganate. It solidifies at 0 Celsius forming shinny orthorhombic crystals with a melting point of 18 Celsius, and a boiling point of 290 Celsius (with decomposition). Glycerol is miscible with water and alcohol, but is insoluble in benzene, chloroform, and carbon disulfide. Glycerol is much less toxic then grain alcohol, making it the least toxic alcohol known. It can be prepared by boiling 50% sodium or potassium hydroxide solution with cow or pig lard. Glycerol can also be obtained by boiling sodium or potassium hydroxide with the lard obtained from cooking bacon. Glycerol can also be prepared from table sugar by fermentation with yeast in the presence of sodium sulfite. Glycerol is prepared on an industrial scale from propylene gas, and is a cheap widely available commercial chemical.

Hexanes.



Hexane

Hexanes are a colorless, very volatile liquid with a faint, peculiar odor. It is rarely sold as n-hexane but usually admixed with hexane isomers simply called "hexanes". Hexane has a boiling point of 69 Celsius, and a melting point of -100 Celsius. It is insoluble in water, but miscible with alcohol, chloroform, and ether. Hexane is a major component of gasoline, and can be distilled from the gasoline using a multiple-path distillation apparatus. Hexane is obtained commercially from petroleum, and is a widely available commercial chemical.



Apparatus for the distillation of unleaded premium gasoline (carefully distill at 70 Celsius).

Hydrochloric acid. *35 - 38% Hydrochloric acid; Water solution of hydrogen chloride*

HCl

Hydrogen chloride

35 – 38% Hydrochloric acid is commonly referred to as concentrated hydrochloric acid. It is a highly corrosive liquid, which evolves choking and corrosive fumes. Some brands of concentrated hydrochloric acid may be colored yellow due to iron. Concentrated hydrochloric acid turns yellow when exposed to sunlight. It forms a constant boiling mixture with water containing 20% hydrogen chloride by weight, and boiling at 108 Celsius. Avoid contact with fumes, and keep in tightly sealed amber glass bottles. Concentrated hydrochloric is prepared on an industrial scale by condensing hydrogen chloride vapors, produced as a by-product in the production of chlorinated hydrocarbons. It can be prepared in the laboratory by mixing sodium

chloride with 98% sulfuric acid and then passing the hydrogen chloride vapors into a quantitative amount of water. Concentrated hydrochloric acid is a widely available commercial chemical. It is available in most hardware stores sold under the name “Muriatic acid”.

40% Hydrofluoric acid

$$HF$$

Hydrofluoric acid forms a colorless highly fuming toxic liquid. It is miscible in water, but is mainly sold as a 40% solution by weight. 40% hydrofluoric acid is obtained by distilling calcium fluoride with concentrated sulfuric acid and dissolving the vapors into water to the correct percentage.

Hydrogen

$$H_2$$

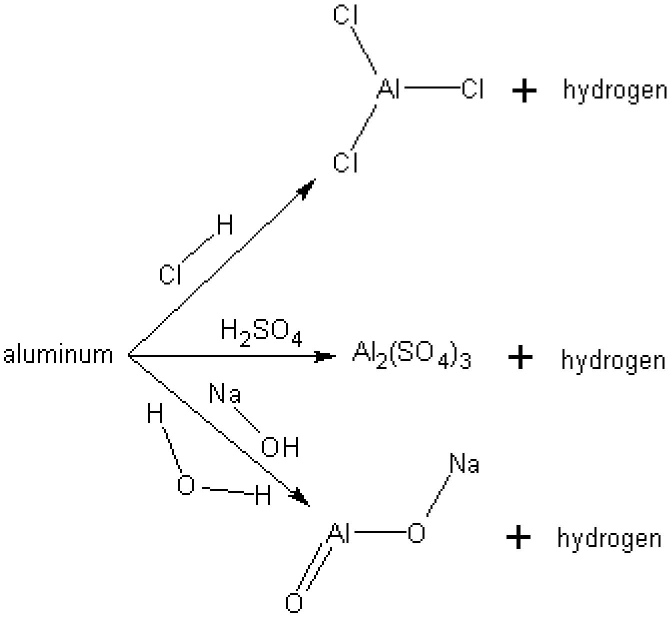
Hydrogen is a colorless, tasteless, highly flammable and explosive gas. Hydrogen is a strong reducing agent when in the presence of a suitable catalyst such as platinum, palladium, nickel, and the like. Hydrogen gas is very difficult to handle and store, but can be stored in compressed cylinders; these cylinders are often hard to purchase and ship, but hydrogen can easily be prepared by reacting dilute hydrochloric acid with zinc, or aluminum, and collecting the resulting hydrogen gas. Note: iron should be avoided as it contains impurities leading to the formation of metal hydrides such as arsine and stibnine, which contaminate the hydrogen (gives the hydrogen a strange metallic like garlic odor). Hydrogen can also be obtained by the electrolysis of acidic or basic water solutions, or by treating a 25% sodium hydroxide (lye) solution with aluminum foil—this latter reaction is utilized by several dry drain opener products to open and clear clogged drains.

Method 1: Preparation of Hydrogen gas from hydrochloric acid or sulfuric acid and a metal, or by reaction of lye with aluminum foil

Materials:

1. 150 grams (5.3 oz.) of concentrated hydrochloric acid	6. 10 grams (0.35 oz.) of aluminum
2. 13 grams (1/2 oz.) of aluminum	7. 17 grams (1/2 oz.) of zinc
3. 45 grams (1.6 oz.) of zinc	8. 50 grams (1.8 oz.) of lye (sodium hydroxide),
4. 75 milliliters (2.5 fluid oz.) of ice cold tap water	9. 150 milliliters of ice-cold tap water
5. 50 grams (1.8 oz.) of concentrated sulfuric acid	10. 11 grams (0.4 oz.) of aluminum (soda pop cans or aluminum foil)

Reaction summary: Hydrogen gas is easily prepared by reacting hydrochloric acid or sulfuric acid with zinc or aluminum, or by reacting lye with aluminum foil. If using hydrochloric acid or sulfuric acid, metal foils or finely divided metals such as aluminum foil, or zinc dust, can give rise to quite violent reactions. For aluminum, aluminum pop cans house the best source, as they are cheap and readily available.



Hazards: Hydrogen is highly flammable and explosive. Mixtures of hydrogen with air when confined will explode when ignited.

Procedure:

Using Hydrochloric acid:

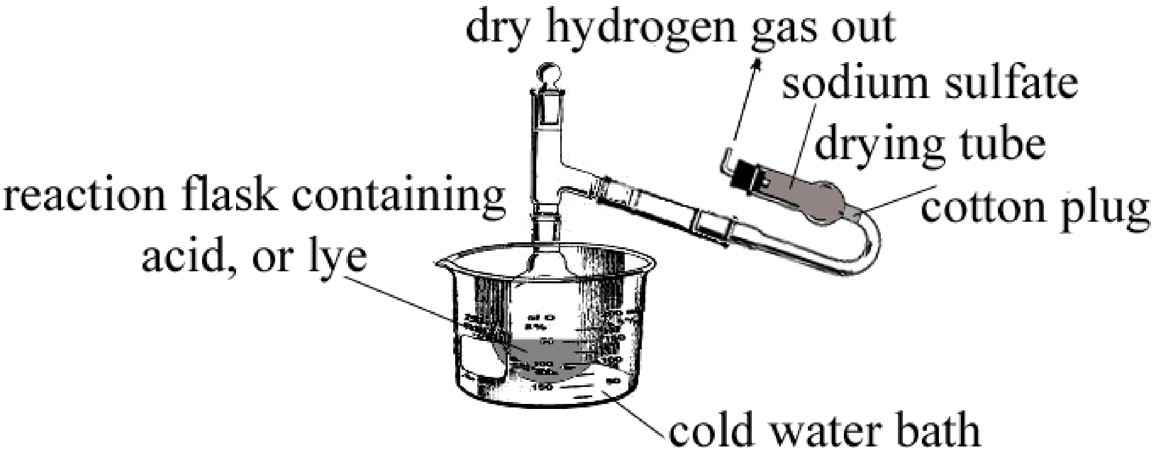
Into the reaction flask of the below illustration, place 150 grams (5.3 oz.) of concentrated hydrochloric acid. Thereafter, slowly add, in small portions at a time, 13 grams (1/2 oz.) of aluminum, preferably aluminum pieces made by carefully cutting-up a clean aluminum can, or 45 grams (1.6 oz.) of zinc. After adding each piece of aluminum or zinc, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

Using Sulfuric acid

Into the reaction flask of the below illustration, place 75 milliliters (2.5 fluid oz.) of ice cold tap water, and then carefully and slowly add in 50 grams (1.8 oz.) of concentrated sulfuric acid. Thereafter, allow the acid mixture to cool (as adding sulfuric acid to water generates excessive heat), and then slowly add, in small portions at a time, 10 grams (0.35 oz.) of aluminum, preferably aluminum pieces made by carefully cutting-up a clean aluminum can, or 17 grams (1/2 oz.) of zinc. After adding each piece of aluminum or zinc, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

Using Lye (sodium hydroxide)

Into a clean beaker, or other suitable glass container, place 50 grams (1.8 oz.) of lye (sodium hydroxide), followed by 150 milliliters of ice-cold tap water. Note: the addition of sodium hydroxide to water generates excessive heat, so allow the solution to cool before using. Thereafter, place the cooled alkaline mixture into the reaction flask of the below illustration. Then slowly add, in small portions at a time, 11 grams (0.4 oz.) of aluminum, preferably aluminum foil pieces. After adding each piece of aluminum, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!



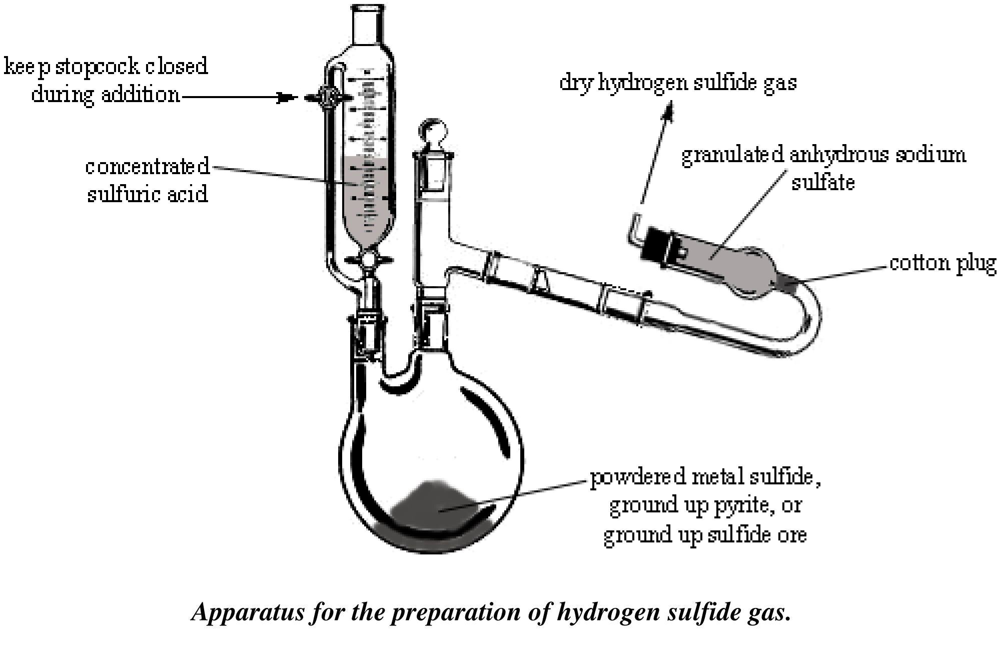
Apparatus for the generation of hydrogen gas.

Hydrogen sulfide

H_2S

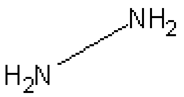
Hydrogen sulfide

Hydrogen sulfide is a flammable, poisonous gas with disagreeable odor of rotten eggs. It can be detected by the human nose in extremely small quantities. Hydrogen sulfide has a sweetish taste. Hydrogen sulfide burns in air with a pale blue flame. It has a melting point of -85 Celsius, and a boiling point of -60 Celsius. Hydrogen sulfide is insoluble in water, and not very soluble in alcohol. It is soluble in glycerol, gasoline, kerosene, carbon disulfide, and crude oil. Hydrogen sulfide is a highly toxic gas, and inhalation can be fatal. Use maximum ventilation when handling. It is evolved from many different natural environmental sources including bacterial decomposition of vegetable and animal proteins, natural springs, natural gas deposits, and volcanoes. Hydrogen sulfide can be obtained from the distillation of petroleum. Hydrogen sulfide is prepared in the laboratory by dropping an acid (usually sulfuric or hydrochloric) onto a metal sulfide such as sodium sulfide, or calcium sulfide (calcium sulfide is prepared by roasting calcium sulfate with charcoal at 1000 Celsius). Hydrogen sulfide is commercially available, but shipping regulations may decrease sale.


keep stopcock closed during addition
dry hydrogen sulfide gas
concentrated sulfuric acid
granulated anhydrous sodium sulfate
cotton plug
powdered metal sulfide, ground up pyrite, or ground up sulfide ore

Apparatus for the preparation of hydrogen sulfide gas.

Hydrazine, anhydrous



Hydrazine

Anhydrous hydrazine forms a colorless liquid that fumes in moist air. The liquid has a penetrating ammonia like odor. The liquid is flammable and burns with a violet flame. The liquid explodes when distilled if air is present; therefore, anhydrous hydrazine should never be distilled in the presence of air. Anhydrous hydrazine has a melting point of 2 Celsius, and a boiling point of 113 Celsius. The liquid is a very powerful reducing agent, and is an excellent solvent for many inorganic compounds. The liquid is very soluble in water forming an azeotrope that contains 68.5% hydrazine by weight, and boiling at 120 Celsius. Anhydrous hydrazine is toxic and ingestion, skin contact, and inhalation of the fumes should be avoided. It is made by dehydrating hydrazine hydrate followed by distilling under nitrogen.

90% Hydrogen peroxide

$$H_2O_2$$

90% hydrogen peroxide forms a water-white colorless slightly syrupy liquid with a melting point of –0.43 Celsius, and a boiling point of 152 Celsius. The liquid decomposes violently when traces of transition metal salts are added or present, and reacts with many organic materials, and in some cases may react violently with combustible materials causing fires. The liquid is miscible with water forming solutions that decompose readily; the liquid is also soluble in ether. 90% hydrogen peroxide can be made in the lab by treating powdered barium peroxide with sulfuric acid, in the presence of a little water, followed by distillation.

99% Hydrogen peroxide

$$H_2O_2$$

99% hydrogen peroxide is a colorless, odorless viscous liquid, which slowly decomposes on standing—99% hydrogen peroxide should be used within 24 hours of preparation. It has a melting point of –0.43 Celsius, and a boiling point of 152 Celsius. It is miscible with water, but is decomposed by many known solvents, and it is a strong oxidizer and contact with combustible material should be avoided. It is obtained by fractional distillation of lower concentrations of hydrogen peroxide, mainly the 90% concentration.

Hydroxylamine

$$NH_2OH$$

Hydroxylamine forms unstable large white flakes, or needles with a melting point of 33 Celsius. The compound is very soluble in water, liquid ammonia, and methanol. The compound decomposes by hot water. Hydroxylamine slowly decomposes when exposed to air, and a sample in a test tube will detonate when heated with a free flame. Hydroxylamine forms salts when dissolved in strong acids. It can be made in the lab by slowly adding small pieces of metallic sodium into liquid ammonia, followed by evaporation, and then recrystallization from methanol.

25 to 30% Hypochlorous acid

$$HOCl$$

25 to 30% hypochlorous acid is a greenish yellow liquid that has no known melting point or boiling point, and solutions slowly decompose, so the acid should be kept in a freezer at –20 Celsius, but should be used within 1 week due to decomposition. Hypochlorous acid is a weak acid that decomposes readily at room temperature, and decomposes rapidly in the presence of sunlight. The 25 to 30% acid can be made by passing moist chlorine gas over mercuric oxide and distilled simultaneously under low vacuum. In the lab, 25 to 30% hypochlorous acid can be prepared by mixing calcium hypochlorite with warm distilled water, followed by suction filtration to remove the calcium hydroxide. Hypochlorous acid is a strong oxidizer, and should be kept away from ammonium chloride at all cost due to the formation of the highly unstable and explosive nitrogen trichloride.

Iodine

$$I_2$$

Iodine forms blackish to purplish-black plates, powder, or granules with a characteristic metallic luster and peculiar odor. It forms a purple vapor when gently heated, and readily volatizes. It has a melting point of 114 Celsius and boiling point of 185 Celsius. Iodine is soluble in aqueous solutions of potassium or sodium iodide, and is soluble benzene, ethyl alcohol, ether, cyclohexane, and methylene chloride. Iodine is capable of producing eye, nose, and throat irritation so wear gloves when handling and use ventilation. Iodine is readily obtainable by bubbling chlorine gas into a solution of potassium or sodium iodide at room temperature, then filtering-off the precipitated iodine, followed by sublimation to purify the iodine. Iodine can also be obtained by treating solutions of potassium or sodium iodide with bleaching powder, potassium permanganate solutions, or with excess bleach (Clorox), then filtering-off the precipitated iodine crystals, followed by sublimation to purify the iodine.

Iron, powdered

Powdered iron forms a grayish black to off black colored powder. When heated in air, it forms the oxide, and it is flammable and burns with a hot flame producing the oxide. Note: powdered iron forms explosive mixtures with oxidizers.

Iron-III-oxide (ferric oxide)

$$Fe_2O_3$$

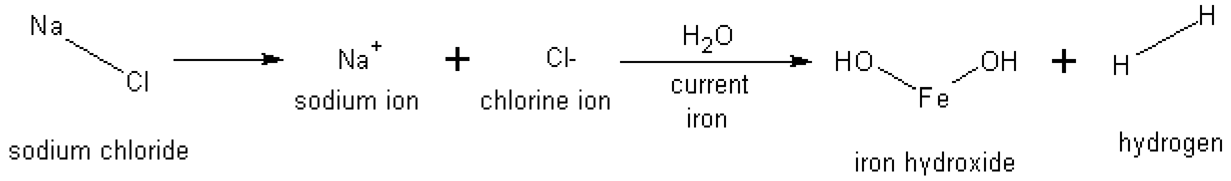
Iron-III-oxide forms a red to reddish to blood red powder with varying chemical composition. It is insoluble in water, and the usual solvents, and iron-III-oxide that has been roasted for several hours is almost insoluble in acids. Iron-III-oxide is a major ingredient in thermite and other incendiary agents.

Method 1: Preparation of Iron-III-oxide from iron nails or rods using an open cell and pickling salt

Materials:

1. 100 grams (3.5 oz.) of pickling salt	3. Two iron nails, or rods ranging 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length
2. 400 milliliters (13.5 fluid oz.) of water	4. 750 milliliters of warm water (25.3 fluid oz.)

Reaction summary: For the preparation of iron-III-oxide, the same electrolysis process is used to form the initial messy mass, and this mass is then collected by filtration and dried in the usual manner. However, instead of drying this mass in a desiccater, it is roasted at high temperature for several hours to facilitate formation of iron-III-oxide, which is formed by the oxidization of the iron-II-oxides.



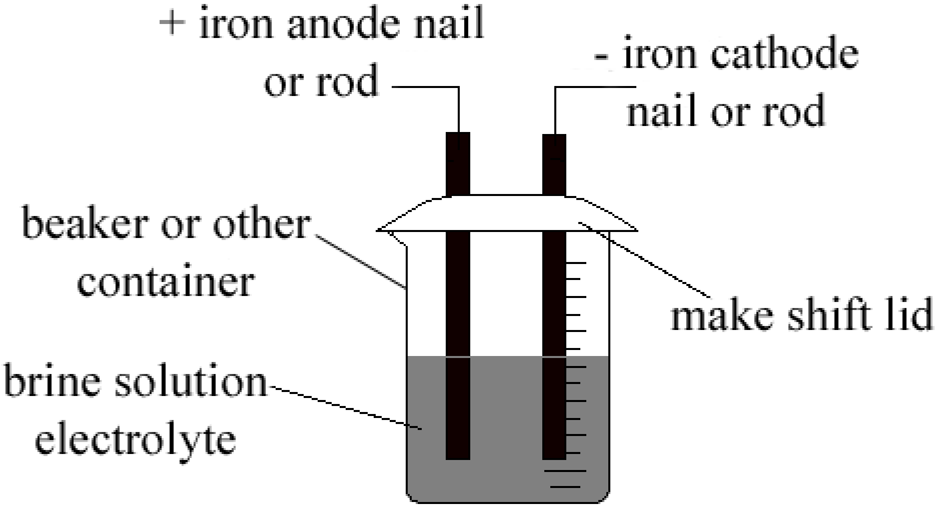
Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated iron oxide slag using an open electrochemical cell

Set-up the apparatus illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two iron nails, or rods ranging 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding nails, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a multi colored precipitate of mixed hydrated iron oxides will precipitate.

The color will vary from green to orange-like. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. The iron anode will slowly corrode and disappear. The electrolysis process can be slow, and can take any where from 12 to 16 hours to complete, depending on the thickness of the iron nails or rods. Thick nails or rods will take much longer to consume as the electro-chemical reaction proceeds. Thin iron nails or rods should be avoided as they will corrode much faster. After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated messy mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the filtered-off mass. Note: the iron nails or rods should be as pure as possible, and steel, galvanized, and plated iron nails or rods should be avoided to avoid the formation of other metal impurities.

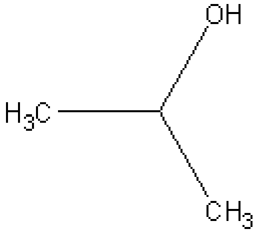


Set-up for the preparation of mixed hydrated iron oxides.

Step 2B: Preparation of iron-III-oxide

To prepare iron-III-oxide, all you need to do is place the dried mass of hydrated iron oxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatized and removed, and the iron oxides are oxidized to iron-III-oxide forming a red powder. After the roasting process, the iron-III-oxide is cooled, and then stored in any suitable container. This iron-III-oxide can be powdered and used in thermite compositions, or pyrotechnics, or used as a catalyst for the oxidation of various gases.

99% Isopropyl alcohol



Isopropyl alcohol is a colorless flammable liquid with a characteristic acetone/ethanol odor. It has a melting point of –88 Celsius, and a boiling point of 82 Celsius. Isopropyl alcohol is miscible in water, alcohol, ether, and chloroform. It is insoluble in salt solutions, and can be extracted from water solutions by the addition of excess salt. Isopropyl alcohol is very common, and is sold under the name “rubbing alcohol”. It is manufactured on a large scale by reacting propylene gas with sulfuric acid followed by treatment with water, and subsequent distillation. To obtain 99% isopropyl alcohol from rubbing alcohol, place 250 milliliters or more of your rubbing alcohol into a separatory funnel, and then add in about 50 to 90 grams of pickling salt. Then shake the entire mixture vigorously for about 10 minutes. Thereafter, allow the separatory funnel to stand for about 10 minutes, and then drain-off the bottom water layer. Note: excess salt will drain out with the water layer. Once the water layer has been removed, place the upper isopropyl alcohol layer into beaker or flask, and then add in 50 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 30 minutes (to absorb moisture). Thereafter, filter-off the magnesium sulfate, and then place the dried filtered isopropyl alcohol into a distillation apparatus, and distill the alcohol at 82 Celsius. When most of the alcohol has distilled over, stop the distillation process and then recover the isopropyl alcohol from the receiver flask. This distilled alcohol will be about 95 to 99% pure isopropyl alcohol, well suitable for use in processes where 99% isopropyl alcohol is needed.

Lead-II-hydroxide

$$Pb(OH)_2$$

Lead-II-hydroxide forms a white heavy poisonous powder. It is insoluble in water, acids, and bases.

25% lithium hydroxide solution

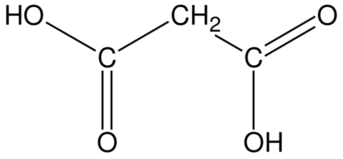
Prepare by carefully adding and dissolving 50 grams of lithium hydroxide into 150 milliliters of water.

Manganese dioxide

$$MnO_2$$

Manganese dioxide forms tetragonal crystals with a rutile structure, forming dark brown to black powder. The powder is insoluble in all known solvents. It is a strong oxidizer and forms explosive mixtures with combustible materials. Manganese dioxide is soluble in hydrochloric acid with evolution of chlorine gas. The compound liberates oxygen when heated. The chief source of manganese dioxide is the mineral pyrolusite.

Malonic acid



Malonic acid

Malonic acid forms tiny colorless crystals or white powder with a melting point of 135 Celsius, with partial decomposition. The compound sublimes under high vacuum. The compound is very soluble in water, and moderately soluble in alcohol, with limited solubility in ether. It is made by reacting monochloroacetic acid with sodium cyanide, followed by reduction of the cyanide group.

Magnesium, and magnesium turnings

$$Mg$$

Magnesium

Magnesium is a slivery-white metal with a melting point of 651 Celsius, and a boiling point of 1100 Celsius. The metal is flammable, and burns with an intense light; even large bars are combustible but require much heat for ignition. Powdered magnesium burns violently and rapidly. The metal is insoluble in water, and only slowly reacts with water forming magnesium hydroxide. The metal readily reacts with dilute acids forming the corresponding salts, and it reacts with ammonium salt solutions forming double salts. The metal is capable of reducing many gases including carbon dioxide, sulfur dioxide, nitric oxide, and nitrous oxide when heated. Magnesium is readily available and can be purchased in many forms and sizes. It is prepared on an industrial scale by the electrolytic reduction of molten magnesium chloride. Magnesium turnings are shiny silver-white to light grayish pieces that tarnish on long exposure to air. The turnings are highly flammable, and absorb carbon dioxide from the air.

Magnesium sulfate, heptahydrate, and anhydrous

$$MgSO_4 – 7H_2O$$

Magnesium sulfate heptahydrate also known as Epsom salt forms colorless crystals that are readily soluble in water, but relatively insoluble in most solvents. The monohydrate occurs in nature as the mineral kieserite. The monohydrate can be converted to the anhydrous form by heating to 250 Celsius for 1 hour. The monohydrate can be prepared by heating the heptahydrate (Epsom salt) to 120 Celsius for 1 hour. The trihydrate forms odorless, colorless crystals. The trihydrtae is converted into the anhydrous salt by heating to 250 Celsius for 1 hour. The trihydrate can be prepared by heating the heptahydrate to 80 Celsius for 1 hour. The heptahydrate occurs in the nature as the mineral epsomite. It forms efflorescent crystals, or a white powder with a bitter, saline, and cool taste. The heptahydrate is soluble in water, but insoluble in alcohol. The heptahydrate is converted to the anhydrous salt by heating to 250 Celsius for 1 hour. The anhydrous salt is highly hygroscopic and absorbs moisture rapidly. The anhydrous salt is a common drying agent, but cannot be used to dry ammonia.

Mercury, liquid

$$Hg$$

Mercury forms a silvery-white, heavy, mobile, liquid metal, which is slightly volatile when strongly heated. Liquid mercury has a melting point of –33.87 Celsius, with a boiling point of 356.7 Celsius. Pure mercury is very stable, and does not tarnish or react with air, but when heated to near its boiling point and passing oxygen gas into it, mercury-II-oxide slowly forms. The metal forms alloys with many metals except iron, it reacts with sulfur when heated. Mercury is a accumulative poison that builds up in the body, but does not cause immediate poisoning when ingested or inhaled until its maximum allowable mass per kg of body weight has been achieved, leading to death. Liquid mercury is made by roasting the mineral cinnabar in a current of dry air.

Mercury-II-oxide

$$HgO$$

Mercury –II-oxide forms a bright red, or orange-red, heavy, odorless, crystalline powder or scales. The product is yellow in color when very finely powdered. Mercury-II-oxide is highly toxic, and caution should be used when handling. The compound decomposes in the presence of light into oxygen and mercury. When heated to 400 Celsius it turns black. The compound decomposes when heated to 500 Celsius. The salt is insoluble in water, but soluble in dilute hydrochloric acid and nitric acid. It is also soluble in solutions of alkali cyanides or iodides.

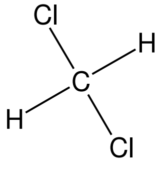
Methyl alcohol. Methanol

$$H_3C\text{——}OH$$

Methanol

Methanol is a flammable, poisonous, and mobile liquid. It has a slight alcoholic odor when pure, and burns with a non-luminous, bluish flame (flames from burning methanol are difficult to see). Methanol has a melting point of –97.8 Celsius, and a boiling point of 65 Celsius. It is miscible with water, alcohol, ether, benzene, acetone, and most organic solvents. Methanol forms azeotropes with many solvents, and it dissolves many inorganic substances. Methanol is a toxic liquid, and ingestion leads to headache, vision problems, and death. The average fatal dose is usually 100 to 200 milliliters, but death has occurred from as little as 30 milliliters. Methanol can be prepared by destructive distillation of wood (heating wood to a high temperature in the absence of air), and then condensing the vapors. The condensed vapors are then distilled to separate the methanol from the acetic acid. Methanol is prepared industrial from carbon monoxide and carbon dioxide. Methanol is a readily available commercial chemical and is the chief ingredient in windshield wiper fluid; from which it can be separated by distillation.

Methylene chloride. *Dichloromethane*



Methylene chloride

Methylene chloride is a colorless, highly volatile liquid, which is insoluble in water, but miscible with ether, and alcohol. It has a boiling point of 40 Celsius, and a melting point of -95 Celsius. Methylene chloride is a powerful, and widely used solvent that is readily available commercially. Methylene chloride is obtained by condensing the vapors obtained by the reaction of dry chlorine with dry methane. The methylene chloride is then separated from the chloroform and carbon tetrachloride by distillation.

99%+ Anhydrous nitric acid (absolute nitric acid)

HNO_3

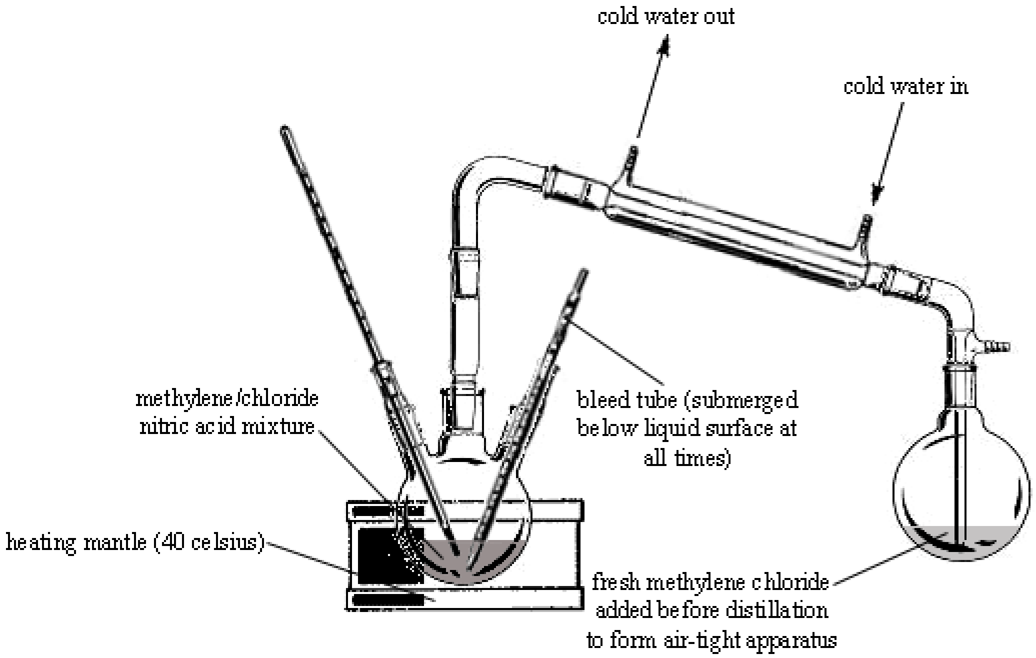
99% nitric acid is a colorless (when freshly prepared), highly fuming, and poisonous liquid. It turns yellow to dark-red on standing, and has a melting point of -41.59 Celsius. 99% nitric acid is a powerful oxidizer. It reacts violently with many substances. 99% nitric acid should be used right after preparation. 99% Nitric acid is commercially available, but shipping regulations restrict its shipment to most locations. *Warning! 99% Nitric acid is very poisonous, and corrosive liquid which evolves large amounts of poisonous fumes. Wear gloves and proper laboratory clothing (lab coat; boots, face shield) when handling this substance and use maximum ventilation.*

Materials:	1. 106 milliliters 70% nitric acid
	2. 94 milliliters 98% sulfuric acid
	3. 1400 milliliters methylene chloride

Summary: 99% Nitric acid can be prepared from 70% nitric acid or any other lower concentration of nitric acid by adding the nitric acid to excess sulfuric acid, and then extracting with methylene chloride. The upper methylene chloride layer is recovered, and then carefully distilled at 40 Celsius to remove the methylene chloride and recover the 99% nitric acid. Commercial & Industrial note: For related, or similar information, see Application No. 549,198, February 12, 1975, by Koppers Company, Inc., to Clifford L. Coon, Menlo Park, CA. Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned substance, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned substance for laboratory, educational, or research purposes.

Hazards: Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.

Procedure: Into an appropriate sized beaker place 94 milliliters of 98% sulfuric, and then 106 milliliters of 70% nitric acid. Then extract this acid mixture with seven 200-millilter portions of methylene chloride. Afterwards, combine all seven portions of methylene chloride (if not already done so). The result will be a 99% nitric acid solution in methylene chloride. This methylene chloride/nitric acid mixture can be used directly in nitrations (if desired), or separated to recover pure 99% nitric acid. To separate the mixture, place the mixture into a distillation apparatus, and then carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. *Note: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might result forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid.*


cold water out
cold water in
methylene/chloride nitric acid mixture
bleed tube (submerged below liquid surface at all times)
heating mantle (40 celsius)
fresh methylene chloride added before distillation to form air-tight apparatus

Apparatus for the distillation of methylene chloride to collect pure nitric acid.

Note: Any concentration of nitric acid can be used instead of just 70% nitric acid. When using nitric acid concentrations below 70% by weight, simply mix the dilute nitric acid with excess sulfuric acid. For example, mix 57 grams of 98% sulfuric acid with 53 milliliters of 60% nitric acid, mix 67grams of 98% sulfuric acid with 53 milliliters of 50% nitric acid, mix 77 grams of 98% sulfuric acid with 53 milliliters of 40% nitric acid, mix 87 grams of sulfuric acid with 53 milliliters of 30% nitric acid, mix 97 grams of 98% sulfuric acid with 53 milliliters of 20% nitric acid, or mix 107 grams of 98% sulfuric acid with 53 milliliters of 10% nitric acid. After one of these mixings, extraction with methylene chloride with seven 100-milliliter portions is the next step. The result is the same as in the above procedure. To isolate the 99% nitric acid, follow the directions in the above procedure.

Alternative method for preparing 99% nitric acid

Summary: 99% Nitric acid can also be obtained by treating potassium or sodium nitrate with sulfuric acid, adding water, and then extracting the acid mixture with methylene chloride to form a solution of 99% nitric acid in methylene chloride. The methylene chloride is then carefully distilled off to recover the pure 99% nitric acid.

Hazards: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might take place forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don’t worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid. Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.

Procedure: Place 194 grams of 98% sulfuric acid into a beaker, and then place the beaker in a ice bath and cool to 0 Celsius by means of an ice bath. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams of potassium nitrate or 84 grams of sodium nitrate over a period of 1 hour while stirring the 98% sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters of cold water while continuously stirring the 98% sulfuric acid mixture and maintaining its temperature at 0 Celsius. Afterwards, remove the ice bath and then extract the acid mixture with seven 150-milliliter portions of methylene chloride. Then combine all seven portions of methylene chloride (if not already done so), and then place the methylene chloride into a distillation apparatus and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask.

90% fuming nitric acid

HNO_3

90% Nitric acid is a yellow to brownish-red, strongly fuming liquid. 90% Nitric acid is a very poisonous and corrosive liquid, which evolves poisonous, choking fumes of nitrogen oxides. It is a powerful oxidizer, and should be kept stored in glass, or Teflon containers, in a cool dry place and away from light (preferably in a refrigerator). 90% Nitric acid reacts violently with many substances. 90% Nitric acid is commercially available, but shipping regulations may limit its sale.

Method of preparing 90% nitric acid

Summary: 90% Nitric acid can be obtained by treating potassium or sodium nitrate with sulfuric acid, mixing with water and then extracting the acid mixture with methylene chloride. The result is a methylene chloride solution containing 99% nitric acid. This methylene chloride/99% nitric acid solution is then mixed with a calculated amount of water, and then distilled to remove the methylene chloride and produce 90% nitric acid.

Hazards: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might take place forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 90% nitric acid. Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.

Procedure: Place 194 grams of 98% sulfuric acid into a beaker, and then place the beaker in an ice bath and cool to 0 Celsius. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams of potassium nitrate or 84 grams of sodium nitrate over a period of 1 hour while stirring the sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters of cold water while continuously stirring the sulfuric acid mixture and maintaining its temperature at 0 Celsius (note: a precipitate may form before or after the addition of the water. If this is happens, never mind it). Then remove the ice bath and then extract the acid mixture with seven 150-milliliter portions of methylene chloride. Afterwards, combine all seven portions of methylene chloride (if not already done so), and then add 7 milliliters of water to the methylene chloride. Then place the entire methylene chloride (two phase) mixture into a distillation apparatus and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask.

70% Nitric acid *Aquafortis; Salpetersaure*

$$HNO_3$$

70% Nitric acid is referred to as concentrated nitric acid, and has a concentration of 65 to 71% nitric acid by weight (simply called 70% nitric acid). 70% nitric acid is a colorless liquid, which fumes in moist air yielding a choking vapor. Its melting point is -41Celsius with a boiling point of 85 Celsius. When boiling 70% nitric acid, it forms a constant boiling mixture with water forming 68% nitric acid with a boiling point of 120 Celsius. 70% nitric acid should be stored in airtight glass bottles away from sunlight. It stains fabrics, and animal tissue a bright yellow. In contact with the skin, it produces a yellow stain. 70% Nitric acid is a powerful oxidizing acid. It reacts violently with many different substances including alcohols, turpentine, charcoal, and organic refuse. 70% Nitric acid begins to turn yellow in contact with strong light sources such as sunlight or magnesium light. 70%Nitric acid is a widely available commercial acid. It is the 7th largest manufactured chemical in the US. Many chemical dealers will not sell 70% nitric acid to unlicensed persons because of its use in manufacturing high explosives. The US government has strong controls over its sale. Although, other methods of obtaining 70% nitric acid are discussed in this manual. *Note: information-right-to-know laws as outlined in the United States Constitution protect the information pertaining to the production of 70% nitric acid discussed in this manual.* Concentrated nitric acid is prepared on an industrial scale by the oxidation of ammonia with air in a special ammonia burner, utilizing platinum as catalyst. *Wear gloves and use proper ventilation when handling 70% nitric acid. 70% Nitric acid can cause skin burns and can chemically react with clothing. Use caution when handling.*

Alternative method for preparing 65 to 70% nitric acid

Summary: 70% Nitric acid can be obtained by treating potassium or sodium nitrate with sulfuric acid, mixing with water, and then extracting the acid mixture with methylene chloride. The result is a methylene chloride solution containing 99% nitric acid. This methlyene chloride/99% nitric acid solution is then mixed with a calculated amount of water, and then carefully distilled to remove the methylene chloride and leave behind 70% nitric acid.

Hazards: methylene chloride/99% nitric acid mixture is very volatile and dangerous. 99% nitric acid is highly toxic, and corrosive. Perform the distillation using proper ventilation, and protect from direct sun light.

Procedure: Place 194 grams of 98% sulfuric acid into a beaker, and then place the beaker in a ice bath and cool to 0 Celsius. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams of potassium nitrate or 84 grams of sodium nitrate over a period of 1 hour while stirring the sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters of cold water while continuously stirring the sulfuric acid mixture and maintaining its temperature at 0 Celsius (note: a precipitate may form before or after the addition of the water. If a precipitate forms, never mind it). Afterwards, remove the ice bath and then extract the acid mixture with seven 150-milliliter portions of methylene chloride. Then combine all seven portions of methylene chloride (if not already done so), and then add 27 milliliters of water to the methylene chloride. Next, place the entire methylene chloride (two phase) mixture into a distillation apparatus and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. *Note: After all the methylene chloride has been removed, the 70% nitric acid may have a slight yellowish to red tint. If this is the case, don't worry. Go ahead and use this nitric acid as 70% nitric acid.*

Nitric oxide

$$NO$$

Nitric oxide is a colorless irritating gas with a boiling point of –151.8 Celsius. It reacts readily with oxygen from the air forming nitrogen dioxide. It is made by reacting copper with 70% nitric acid in an inert atmosphere.

Nitrogen

$$N_2$$

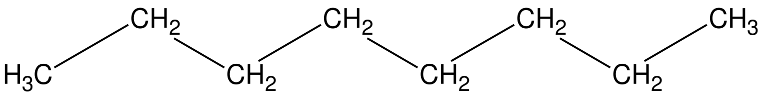
Nitrogen is a colorless, tasteless, odorless gas that comprises 75.5% of the earth's atmosphere. The gas has a boiling point of –195.79 Celsius, and a melting point of –210 Celsius. Nitrogen gas is relatively inert and only reacts with other elements under extreme conditions. It can be prepared in the lab be heating sodium azide. Nitrogen is a very inexpensive gas sold under numerous brand names and as a liquid.

Nitrogen dioxide

$$NO_2$$

Nitrogen dioxide forms a brownish reddish gas that is very toxic and highly irritating to the eyes, nose, and throat. The gas readily liquefies when cooled to 21 Celsius. Nitrogen dioxide has a melting point of –9.3 Celsius, and a boiling point of 21 Celsius. Nitrogen dioxide is prepared on a commercial scale by the oxidation of ammonia with oxygen over heated platinum. In the lab, nitrogen dioxide can be made by heating lead-II-nitrate in a sealed apparatus, or by reacting copper with 70% nitric acid in the presence of air.

n-Octane



n-Octane

Octane forms a colorless volatile highly flammable liquid with a melting point of –56.8 Celsius, and a boiling point of 125.6 Celsius. Octane is slightly soluble in alcohol, but very soluble and miscible with benzene, toluene, and petroleum ether. Octane is a major component of gasoline and is added as a anti-knocking agent. It is obtained from the distillation of petroleum, but can be obtained from multi path distillation of gasoline, see vide supra hexane.

Osmium tetraoxide

$$OsO_4$$

Osmium tetraoxide forms a pale yellow solid, forming monoclinic crystals. Warning! Very poisonous, handle with extreme care. The dry solid has an acrid chlorine like odor, with a melting point of 40.6 Celsius, and a boiling point of 130 Celsius;

however, begins to sublime before reaching its melting point temperature. Osmium tetraoxide is soluble in benzene, ether, alcohol, aqueous ammonia solution, and only slightly soluble in water. The solid can be made by passing dry oxygen over powdered osmium in a sealed tube.

Oxygen difluoride

$$F_2O$$

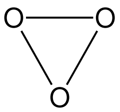
Colorless stable gas or yellowish-brown liquid with a peculiar odor. Does not attack glass in the cold, but will slowly attack glass when heated. Reacts only very slowly with water, and is insoluble in most solvents. The gas has a melting point of –233.8 Celsius, and a boiling point of –145 Celsius. It is made by passing fluorine gas into distilled water at low temperature in a special nickel apparatus.

Oxygen gas

$$O_2$$

Oxygen is a colorless, odorless, tasteless gas, which comprises 20.95% of the earth’s atmosphere, and 46.6% of the earths crust. Oxygen has a melting point of –218.4 Celsius, and a boiling point of –182.96 Celsius. Liquid oxygen is collected by fractional distillation of liquid air. Oxygen is by far the most important element on earth, and is necessary for combustion.

Ozone



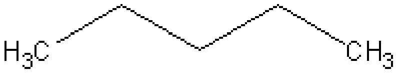
Ozone

Ozone forms a bluish, explosive, highly pungent and irritating gas, or deep blue liquid (at very cool temperatures). Ozone gas is the main component of the upper-upper atmosphere that gives the sky its bluish color and protects earth’s surface from harmful space radiation. The gas is made by passing oxygen through a high voltage AC tube, followed by immediate cooling to liquefy the ozone. Ozone is a powerful oxidizing agent, with a melting point of –193 Celsius, and a boiling point of –111.9 Celsius. Warning! Solutions of ozone explode when warmed.

Palladium foil

Palladium foil is a shiny silvery-white metal similar to aluminum foil, but it is very inert to chemical attack. It has a melting point of 1555 Celsius, and reacts slowly with concentrated hydrochloric and sulfuric acids.

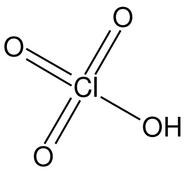
Pentanes



Pentanes

Pentanes are a highly flammable liquid with a melting point of –129 Celsius, and a boiling point of 36 Celsius. Pentanes are insoluble in water, but soluble in many organic solvents. Pentanes can be obtained in a similar manner as for hexanes, see vide supra, and can be used in place of hexanes.

98% perchloric acid



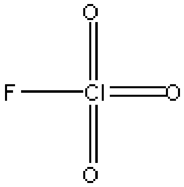
Perchloric acid

Perchloric acid forms a colorless, volatile, very hygroscopic liquid. The acid has a boiling point of 19 Celsius under 19mm of mercury. Note: distilling perchloric acid at atmospheric pressure can lead to explosion. Perchloric acid combines with water with generation of great heat. 98% acid is not commercially available due to the dangers of violent decomposition, and it is marketed at concentrations ranging from 60 to 70% by weight. 98% perchloric acid should be used within 24 hours of preparation, and it should be stored during this 24-hour period in a freezer. 98% perchloric acid is a dangerous substance and should only be handled when using a blast shield with proper conditions. It can explode in contact with many organic materials so use extreme caution. It is prepared by carefully high vacuum distillation of sodium perchlorate with sulfuric acid.

70% perchloric acid

70% perchloric acid is commercially available, and forms a colorless slightly fuming volatile liquid. It is much safer to use then the 98% concentration, and it is made by reacting potassium perchlorate with sulfuric acid, followed by dilution with water to 70% concentration, and during the dilution process potassium sulfate will precipitate, and can then be filtered-off to collect the 70% acid.

Perchloryl fluoride



Perchloryl fluoride

Perchloryl fluoride is a non corrosive gas with a sweet taste. It has a melting point of -148 Celsius and a boiling point of -47 Celsius. Perchlorly fluoride is a very stable gas at room temperature. Explosions will result if perchloryl fluoride is treated with reducing agents such as amides, powdered metals, hydrides, sulfites, phosphites, phosphides, or hydrazines or when treated with alcohols. Avoid skin contact and inhalation. It is prepared by careful distillation of a mixture of antimony pentafluoride and potassium perchlorate. A series of condensers is used and the end result is the perchloryl fluoride is condensed using a liquid nitrogen cold trap.

Phosphine

$$PH_3$$

Phosphine is a highly toxic colorless fishy smelling gas, which is spontaneously combustible in air. The gas has a boiling point of –87.7 Celsius, and a melting point of –133 Celsius. The gas reacts violently with oxygen and the halogens. Forms phosphides when passed over heated metals. Forms salts when mixed with halogen acids. Phosphine is primarily made by acid hydrolysis of metal phosphides with sulfuric acid; for example dripping concentrated sulfuric acid onto calcium phosphide in an inert atmosphere.

Phosphorus, red

$$P_4$$

Red phosphorus forms a red to violet powder. The properties of red phosphorus are intermediate between white phosphorus and black phosphorus. The element sublimes when heated to 416 Celsius. The powder is insoluble in all known solvents, except phosphorus tribromide. Red phosphorus is much less reactive then white phosphorus, and does not react with other elements or compounds under moderate conditions. Forms white phosphorus when distilled at 290 Celsius under high vacuum. It ignites spontaneously when heated in air to 260 Celsius burning to the pentoxide.

Phosphorus pentachloride

$$PCl_5$$

Phosphorus pentachloride forms white to pale yellow, fuming, very deliquescent crystalline mass with a pungent unpleasant odor that is highly irritating. The compound has a melting point of 148 Celsius under reduced pressure, and a boiling point of 160 Celsius. The compound reacts vigorously with water and alcohols. Phosphorus pentachloride is soluble in chloroform and carbon disulfide. It is made by chlorination of phosphorus trichloride in chloroform at 100 Celsius in a reflux apparatus under an inert atmosphere, followed by distillation at 160 Celsius under an inert atmosphere.

Phosphorus trichloride

$$PCl_3$$

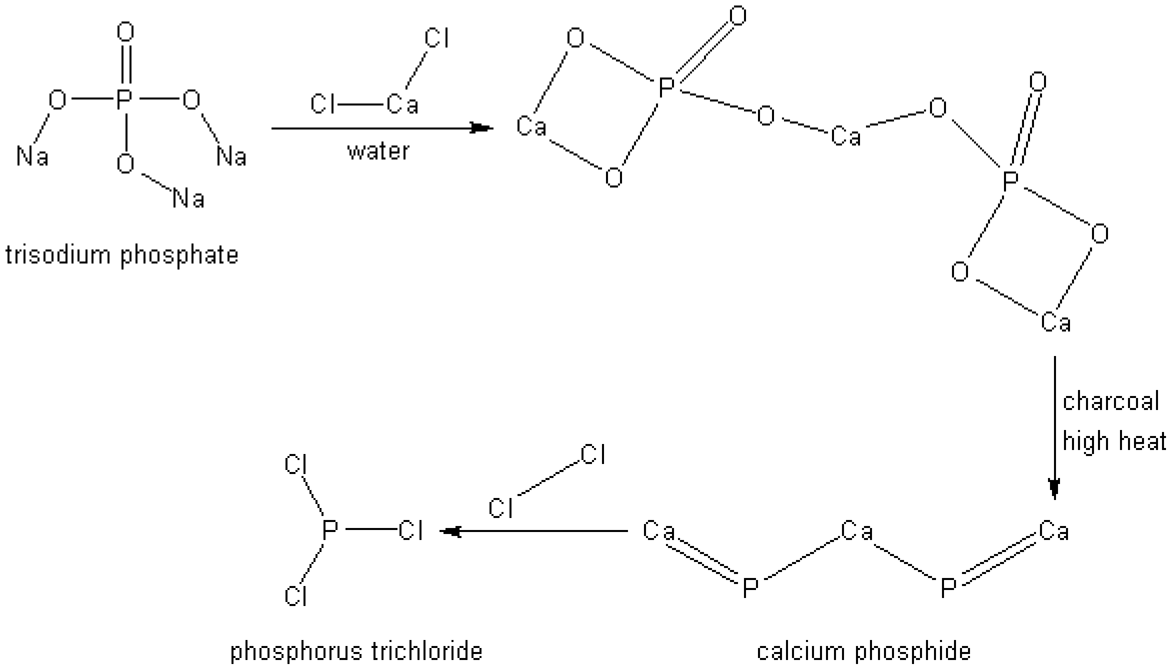
Phosphorus trichloride forms a colorless to slightly colored fuming liquid with a melting point of –112 Celsius, and a boiling point of 76 Celsius. The liquid is highly reactive and decomposes rapidly in the presence of water or alcohol. The liquid should be kept in airtight bottles and stored in a cool dry place. It is soluble in benzene, methylene chloride, ether, and hexane. Avoid inhalation of the fumes and skin contact.

Method 1: Preparation of Phosphorus trichloride from trisodium phosphate, chlorine, calcium chloride, and charcoal

Materials:

1. 250 grams (8.8 grams) of technical grade (store bought) trisodium phosphate	6. 100 grams (3.5 oz.) of finely powdered wood charcoal
2. 750 milliliters (25.2 fluid oz.) of warm water	7. 250 milliliters (6.9 fluid oz.) of methylene chloride
3. 250 grams (8.8 oz.) of anhydrous calcium chloride	8. 200 grams (7 oz.) of extra dry chlorine gas
4. 750 milliliters (25.2 fluid oz.) of cold water	9. 100 grams (3.5 oz.) or so of aluminum oxide
5. 1500 milliliters (50.7 fluid oz.) of warm water	

Reaction summary: Phosphorus trichloride can be prepared in a three-step process starting with the formation of calcium phosphate. This crude technical grade of calcium phosphate is prepared by mixing aqueous solutions of trisodium phosphate and calcium chloride. The mixing causes the water insoluble calcium phosphate to precipitate. The precipitate is readily filtered-off, washed, and then dried in the usual manner. Thereafter, the crude calcium phosphate is then roasted at high temperature in the presence of carbon, whereby it gets reduced to calcium phosphide. Note: some elemental phosphorus may be liberated in the reaction, so use proper ventilation. The calcium phosphide crude mixture is then chlorinated to form a mixture of compounds, one of which being the desired phosphorus trichloride. After the chlorination process, the reaction mixture is filtered, and then distilled using a conventional distillation apparatus to remove the methylene chloride solvent. Finally, the remaining crude liquid left over after removal the methylene chloride, is then fractionally distilled to recover the liquid product of phosphorus trichloride. The distilled phosphorus trichloride can be re-distilled if desired.



Hazards: Use good ventilation when working with chlorine gas, and avoid inhalation of the fumes. Use caution when heating the calcium phosphate as small amounts of white phosphorus may be evolved.

Procedure:

Step 1: Preparation of calcium phosphate

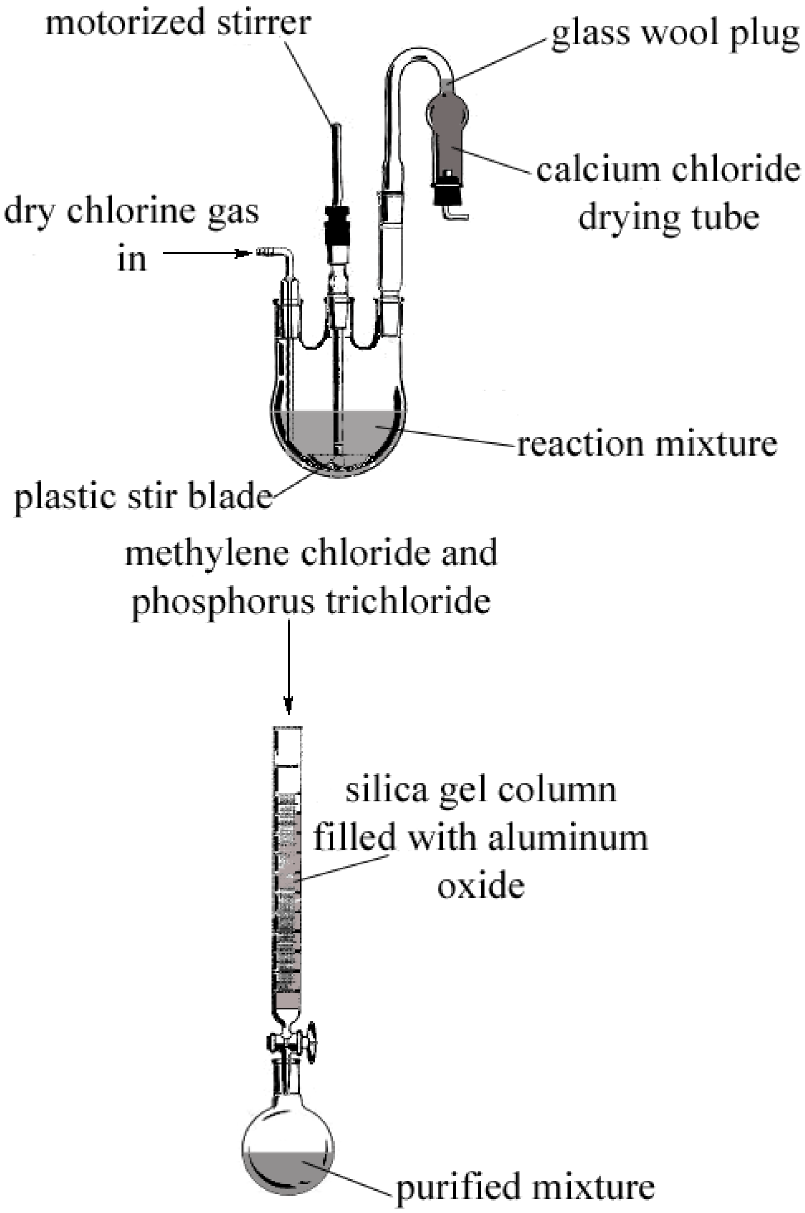
Into a suitable beaker or similar container, place 250 grams (8.8 grams) of technical grade (store bought) trisodium phosphate, and then add in 750 milliliters (25.2 fluid oz.) of warm water, and then stir the mixture to dissolve all of the trisodium phosphate (technical grade). Note: technical grade and store bought trisodium phosphate (TSP) contains sodium carbonate and maybe some sodium hydroxide to prevent the product from caking. This sodium carbonate and any other alkaline product needs to be neutralized before the initial reaction. To do this, first, place 250 grams (8.8 grams) of the dry TSP product from the packaging container into a suitable crucible or similar container, and then roast it at 150 to 250 Celsius for 1 hour to remove several moles of water of hydration. Thereafter, break-up the re-solidified TSP product from the crucible or similar container, and then dissolve it into 750 milliliters (25.2 fluid oz.) of water contained in a suitable beaker or container. Thereafter, drip hydrochloric acid, battery acid, or sulfuric acid into the trisodium phosphate solution until the evolution of carbon dioxide gas ceases. Once this point is achieved, the sodium carbonate has been neutralized. Now, to this trisodium phosphate solution, add in a calcium chloride solution prepared by adding and dissolving 250 grams (8.8 oz.) of anhydrous calcium chloride into 750 milliliters (25.2 fluid oz.) of cold water—heat will be evolved upon dissolving. Note: anhydrous calcium chloride is obtained by placing a sample of at least 300+ grams of technical grade calcium chloride, available in hardware stores and grocery stores sold as “ice melter”, into a crucible or similar container, and then roast the technical grade calcium chloride at 200+ Celsius to remove the water of hydration. Thereafter, the anhydrous calcium chloride can be dissolved into the 750 milliliters (25.2 fluid oz.) of water, once the anhydrous salt has cooled. Upon mixing the two solutions of trisodium phosphate and calcium chloride, a precipitate of crude calcium phosphate tribasic, mixed with a small amount of other calcium phosphates, will form a chunky and messy precipitate. Thereafter, filter-off this chunky precipitate using gravity filtration, or preferably vacuum filtration, and then wash this precipitate with three 500 milliliter (three 16.9 fluid oz. portions) portions of warm water. Thereafter, vacuum dry or air-dry the washed filtered-off precipitate. Finally, place the dried calcium phosphate into a crucible or similar container, and then roast it at 100 to 150 Celsius for an hour or so to remove any moisture or water of hydration. Thereafter, pulverize the heated calcium phosphate into a fine powder, once it has cooled.

Step 2: Preparation of calcium phosphide

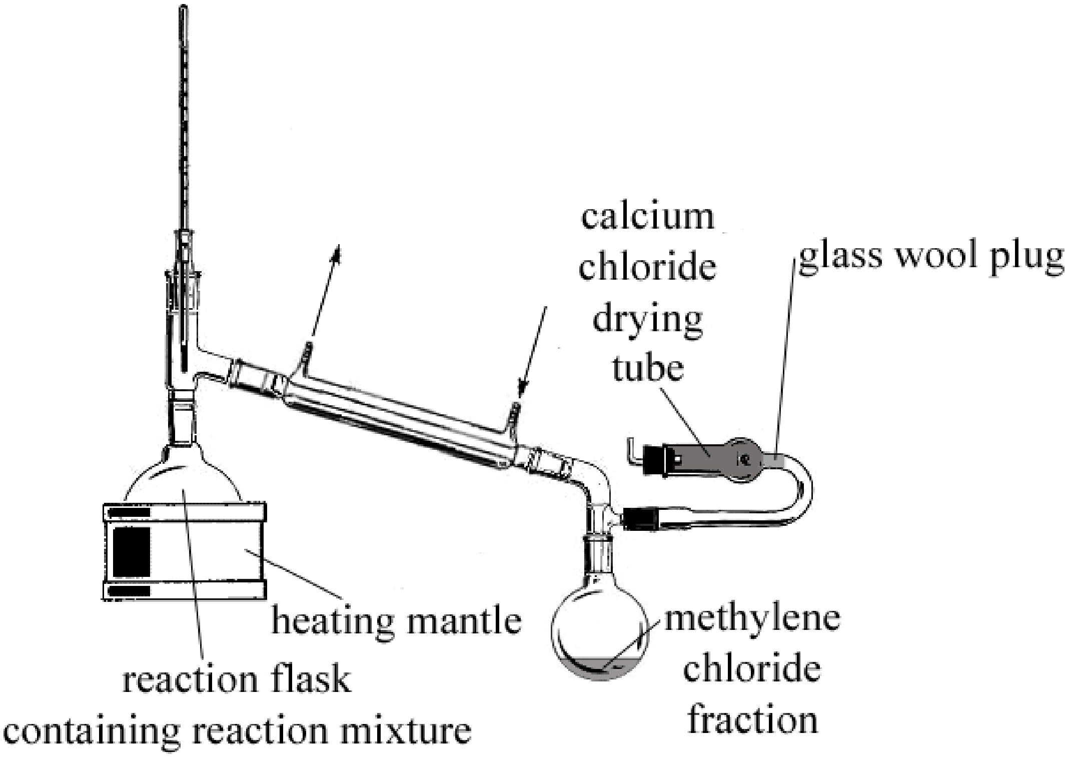
Now, into a zip lock bag, or similar plastic container, place the pulverized calcium phosphate (prepared in step 1), followed by 100 grams (3.5 oz.) of finely powdered wood charcoal (average charcoal used for cooking, with no quick-light garbage or other chemical addictives), and then shake the bag thoroughly for about 10 to 15 minutes to thoroughly mix both solids. Thereafter, place this mixed powdered mixture into any high heat resistant crucible, such as stainless steel crucible, nickel, or quartz crucible, or equivalent, and then blast this mixture at 1000 Celsius using a high temperature Meeker Bunsen burner or equivalent for about 1 hour or so. If a high temperature Bunsen burner is unavailable, you can use a fire or some other means of heat. After the roasting process, remove the heat source, and allow the hot mixture to cool to room temperature. Now, at this point, the blackish-gray mixture will be composed of calcium phosphide, calcium oxide, a little calcium carbide, unreacted calcium phosphate, and un-reacted charcoal. Keep this mixture as is for use in step 3.

Step 3: Preparation of phosphorus trichloride

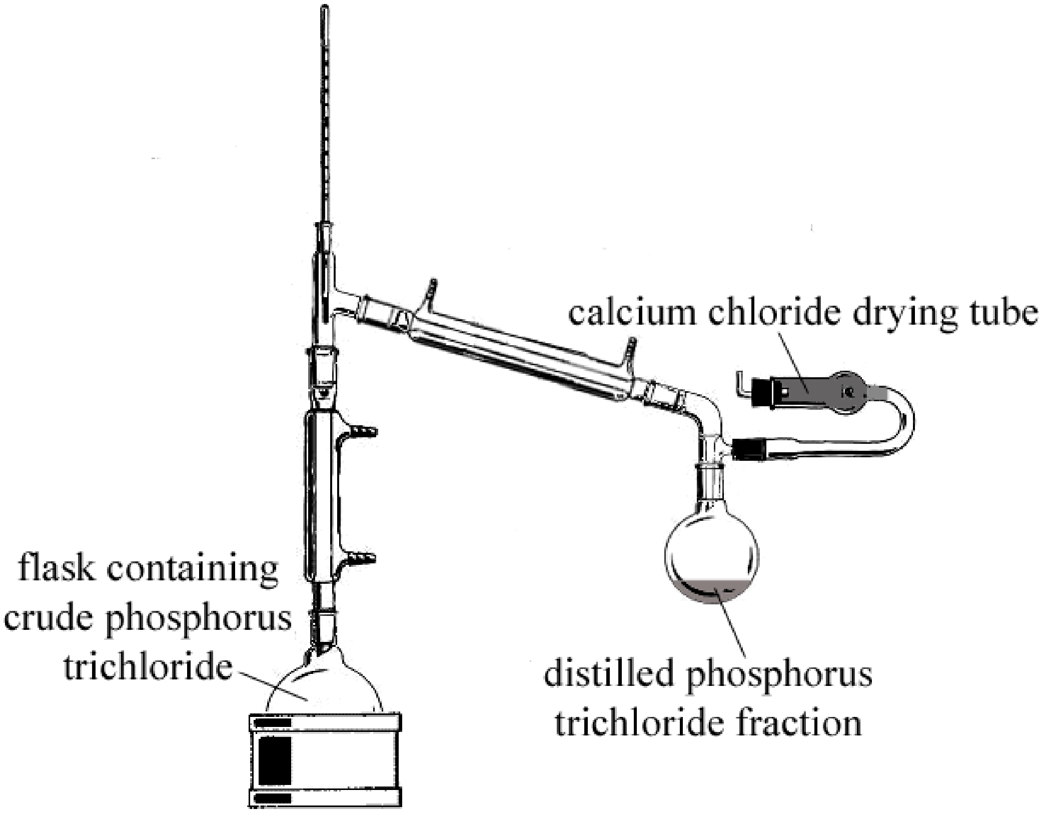
Into a suitable apparatus, as illustrated below, place the crude product obtained in step 2, and then add in 250 milliliters (6.9 fluid oz.) of methylene chloride. Then begin the motorized stirrer on moderate speed, and then bubble into the mixture, 200 grams (7 oz.) of extra dry chlorine gas over a period of 2 to 6 hours while stirring the reaction mixture on moderate speed. Note: dry chlorine can be obtained by passing the chlorine through multiple anhydrous calcium chloride drying tubes. During the addition of the chlorine gas, phosphorus trichloride will form, and will be taken-up into the methylene chloride. The by-products, will form insoluble precipitates along with the carbon. After the addition of the chlorine gas, the mixture needs to be filtered, to remove the insoluble materials, and then the resulting filtered mixture needs to be passed several times, through a silica gel column, filled with 100 grams (3.5 oz.) or so of aluminum oxide (see illustration). Thereafter, the mixture should be placed in a distillation apparatus, and first, distilled at 40 Celsius to remove the methylene chloride. Thereafter, place the remaining mixture into a fractional distillation apparatus, and fractionally distill the product mixture at 76 Celsius to collect the phosphorus trichloride. Note: during all distillations, keep a drying tube attached to the apparatus to keep moisture out.


motorized stirrer
glass wool plug
calcium chloride drying tube
dry chlorine gas in
reaction mixture
plastic stir blade
methylene chloride and phosphorus trichloride
silica gel column filled with aluminum oxide
purified mixture

Top illustration: Apparatus for the addition of chlorine gas to a mixture of methylene chloride and calcium phosphide. Bottom illustration: setup for the purification of the methylene chloride reaction mixture containing the phosphorus trichloride. The aluminum oxide helps remove impurities, and carbon.

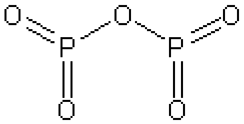

calcium chloride drying tube
glass wool plug
heating mantle
methylene chloride fraction
reaction flask containing reaction mixture

Distillation apparatus for removal of the methylene chloride.


calcium chloride drying tube
flask containing crude phosphorus trichloride
distilled phosphorus trichloride fraction

Fractional distillation apparatus for distillation of phosphorus trichloride.

Phosphorus pentoxide



Phosphorus pentoxide

Phosphorus pentoxide forms white very deliquescent crystals that are very corrosive. The solid material has a melting point of 340 Celsius, but sublimes when heated to 360 Celsius. The solid material is very hygroscopic and reacts with water and alcohol forming phosphoric acid. Phosphorus pentoxide is a non-oxidizing solid that does not support combustion. It is prepared by burning white phosphorus in a current of dry air and condensing the vapors.

Platinized asbestos

Platinized asbestos is made by taking woven asbestos wool, wetting it with a little ether, and then sprinkling finely divided platinum onto the wool, and then drying.

Platinum dioxide

PtO_2

Platinum dioxide is also known as “Adams catalyst”, and is a jet-black powder that is insoluble in acids and bases, but when freshly prepared it can dissolve into strong acids and bases. Platinum dioxide is made by reacting a water solution of platinic chloride with sodium hydroxide solution, followed by suction filtration, washing, and drying.

Platinum gauze

Platinum gauze resembles steel wool but has a brilliant silvery white appearance. The gauze is very very expensive, but if used properly can be re-used over and over again.

Potassium, metallic

Metallic potassium forms a brilliant silvery-white metal with a body-centered cubic structure. It rapidly tarnishes on exposure to air, and like metallic sodium should be stored submerged in kerosene. The element has a melting point of 63 Celsius, with a boiling point of 762.5 Celsius. Metallic potassium is soluble in liquid ammonia forming salts. The element reacts violently with water producing explosions, and it reacts vigorously with many different compounds. Metallic potassium is a powerful reducing agent, and can reduce nitrates, sulfates, phosphates, and others. It is made my electrolysizing molten potassium hydroxide.

Potassium dichromate

$K_2Cr_2O_7$

Potassium dichromate forms beautiful bright orange-red crystals, which are not hygroscopic or deliquescent. The salt is a strong oxidizer and forms explosive mixtures with combustible materials. It has a melting point of 398 Celsius, but decomposes when heated to 500 Celsius. Potassium dichromate is moderately soluble in water, but insoluble in most known solvents. It is made by fractional crystallization of a saturated solution of sodium dichromate with a saturated solution of potassium hydroxide, or made by roasting chrome ore with potassium hydroxide.

Potassium hydroxide

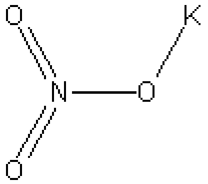
KOH

Potassium hydroxide forms white or slightly yellow lumps, rods, or pellets, which rapidly absorb moisture and carbon dioxide from the air. It has a melting point of 360 Celsius. Pure potassium hydroxide has a melting point of 380 Celsius. It is very soluble in water, alcohol, and glycerol generating much heat when dissolved. Potassium hydroxide, and its solutions are very corrosive to the skin, and can produce burns. Keep dry potassium hydroxide or its solutions in tightly sealed bottles. Potassium hydroxide is toxic and ingestion causes tissue damage. It is prepared by the same manner as sodium hydroxide, and is a widely available commercial chemical.

50% potassium hydroxide solution

Easily prepared by dissolving 100 grams of potassium hydroxide into 100 milliliters of water.

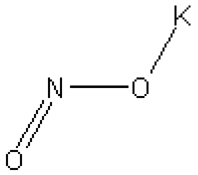
Potassium nitrate



Potassium nitrate

Potassium nitrate forms colorless transparent prisms, or white granules or crystalline powder. The crystals have a melting point of 333 Celsius, but begin to decompose when heated to 400 Celsius, with evolution of oxygen. The compound is somewhat soluble in water, but insoluble in alcohol, and most common organic solvents. Potassium nitrate is widely used in pyrotechnic compositions, and is made by fractional crystallization of sodium nitrate and potassium hydroxide.

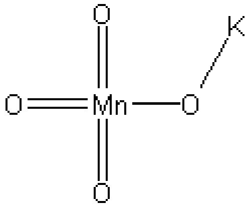
Potassium nitrite



Potassium nitrite

Potassium nitrite forms white to slightly yellowish deliquescent granules or powder. The crystals readily decompose when treated with mineral acids forming brown fumes. The crystals have a melting point of 441 Celsius, with decomposition beginning at 350 Celsius. Potassium nitrite is readily soluble in water, but has low solubility in alcohol and most organic solvents. The article of commerce usually contains about 85% nitrite, with the nitrate making up the rest.

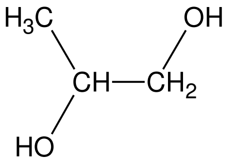
Potassium permanganate



Potassium permanganate

Potassium permanganate forms dark purple or bronze-like, odorless crystals, which are stable in air. Potassium permanganate decomposes when heated at 240 Celsius with evolution of oxygen. It is not very soluble in cold water but is more soluble in warm or hot water. It reacts with alcohol and many other organic solvents, and hence, is a powerful oxidizer. Potassium permanganate reacts with hydrochloric acid liberating chlorine, and it forms explosive mixtures with all combustible materials. Solutions of potassium permanganate have an intense purple color. Handle potassium permanganate with care. Potassium permanganate is prepared from manganese ore. It is a common and commercially available chemical.

Propylene glycol



Propylene glycol

Propylene glycol forms hygroscopic, viscous liquid, with a slight acrid taste and a melting point of –59 Celsius, and a boiling point of 188 Celsius. The viscous liquid is miscible with water, acetone, and chloroform. It is also soluble in ether and will dissolve many essential oils. Propylene glycol is non-toxic and is used in the food industry, and also used as an antifreeze for automobiles. It is made by hydrolysis of propylene oxide.

Sodium azide

NaN_3

Sodium azide forms colorless hexagonal crystals, which decompose on heating into metallic sodium and nitrogen. This decomposition often occurs violently. Sodium azide is very soluble in water, and water solutions are rapidly converted to hydrazoic acid. Sodium azide solutions should not be stored for prolonged periods of time. Sodium azide is insoluble in ether, and liquid ammonia. It is a poisonous solid, and ingestion of 600 to 800 milligrams may be fatal. Keep sodium azide in tightly sealed bottles, and store in a cool dry place away from light. It can be prepared by reacting heated nitrous oxide (60 to 90 Celsius) with sodium amide heated at 60 to 100 Celsius, and then recrystallizing the sodium azide from water. Sodium azide is commercially available, but expensive.

Sodium, metallic

Na

Metallic sodium forms light, silvery-white rods with a body-centered cubic structure. The metal is a brilliant silvery white color when freshly prepared or cut, but tarnishes rapidly in air. Metallic sodium has a melting point of 97.8 Celsius, with a high boiling point of 881 Celsius. The metal explodes in contact with water, and reacts vigorously with alcohols, carboxylic acids, and many other organic materials. Metallic sodium is prepared on an industrial scale from electrolysizing molten sodium hydroxide using a graphite cathode, and a steel anode, and then collecting the molten sodium, which floats on the surface of the molten sodium hydroxide. In the lab, metallic sodium can be prepared by heating sodium azide.

Sodium bicarbonate

$NaHCO_3$

Sodium bicarbonate forms a white powder or granules. When heated to 100 Celsius, it breaks down into anhydrous sodium carbonate. The compound readily decomposes by acids forming sodium salts. Sodium bicarbonate is made by saturating sodium hydroxide solution with an excess of carbon dioxide, followed by suction filtration, washing, and drying. Sodium bicarbonate is also known as baking soda.

5% baking soda solution

Prepare by dissolving 50 grams of baking soda into 950 milliliters of water.

Sodium bromide

NaBr

Sodium bromide forms colorless to white crystalline powder, or granules with a melting point of 755 Celsius. Sodium bromide is soluble in water, with moderate solubility in alcohol. It forms a dihydrate when dissolved in water forming cubic crystals upon evaporation of the water. Sodium bromide reacts with chlorine liberating the red liquid elemental bromine.

Sodium carbonate, and anhydrous sodium carbonate

$$Na_2CO_3$$

Anhydrous sodium carbonate is also called *Solway soda*, or *soda ash*. It is a odorless, hygroscopic powder with a melting point of 851 Celsius (begins to lose carbon dioxide when heated at 400 Celsius forming sodium oxide). Anhydrous sodium carbonate absorbs moisture from the air. It is soluble in glycerol and water, but insoluble in alcohol. It decomposes by acids with violent liberation of carbon dioxide. It combines with water evolving heat, and water solutions are strongly alkaline. The monohydrate forms odorless, small crystals or crystalline powder, which becomes anhydrous when heated at 100 Celsius. It is soluble in water and glycerol, but insoluble in alcohol. The decahydrate also called *Nevite*, or *washing soda*, forms transparent crystals, with a melting point of 34 Celsius. It is soluble in water and glycerol, but insoluble in alcohol. The decahydrate occurs in nature as the minerals *thermonatrite*, and *natron*. Anhydrous sodium carbonate is a widely available commercial chemical. It can be prepared by directly heating baking soda to 300 or 400 Celsius for one hour, or from scratch by bubbling carbon dioxide gas through a solution of sodium hydroxide, followed by filtration to remove the bicarbonate (baking soda), which is then roasted at 300 to 400 Celsius for one hour.

5% sodium carbonate solution

Prepare by dissolving 5 grams of sodium carbonate into 95 milliliters of water.

10% sodium carbonate solution

Prepare by dissolving 50 grams of anhydrous sodium carbonate into 450 milliliters of water.

15% sodium carbonate solution

Prepare by dissolving 50 grams of anhydrous sodium carbonate into 282 milliliters of water.

Sodium chloride

$$NaCl$$

Sodium chloride also known as common table salt or rock salt forms colorless cubic crystals, or white powder or granules. The salt has a melting point of 804 Celsius, and it begins to volatize when heated to higher temperatures. The salt is soluble in water, and glycerol, but relatively insoluble in most solvents.

Sodium fluoride

$$NaF$$

Sodium fluoride forms tetragonal or cubic white or colorless crystals with a melting point of 993 Celsius. Sodium fluoride is soluble in water, but insoluble in alcohol, and many organic solvents. The aqueous solution etches glass, and hence should not be stored for prolonged periods of time in glass containers. The dry solid can be stored in glass bottles. Sodium fluoride is available in many household pesticides formulations, and can be made by neutralizing sodium hydroxide with hydrofluoric acid.

Sodium hydroxide

$$NaOH$$

Sodium hydroxide

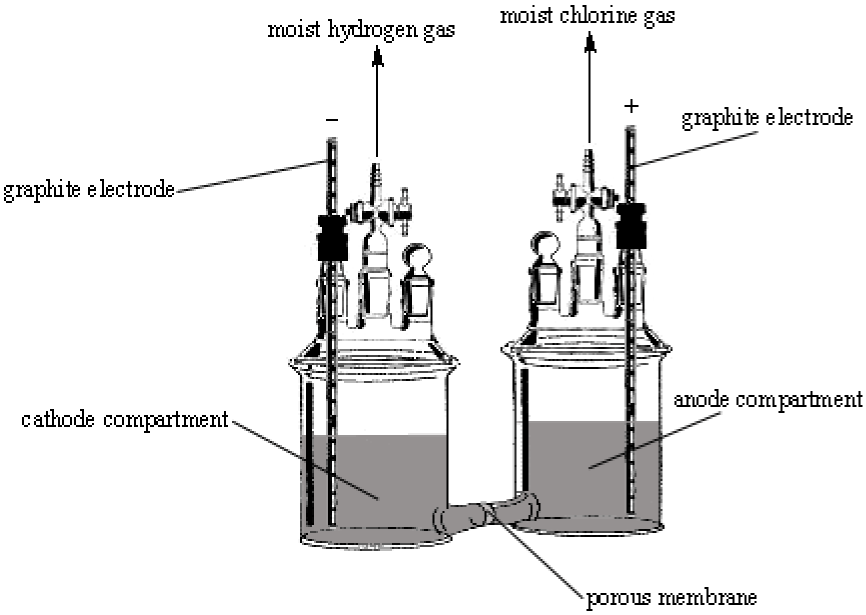
Sodium hydroxide forms fused solid pieces, granules, rods, or powder. It rapidly absorbs moisture and carbon dioxide from the air. Solutions of sodium hydroxide are very corrosive to animal tissue, and aluminum. It has a melting point of 318 Celsius. Sodium hydroxide is very soluble in water and alcohol. It generates large amounts of heat when dissolving in water, or when mixed with acid. Sodium hydroxide is toxic. Handle sodium hydroxide with care. Sodium hydroxide is a widely available commercial chemical, which is sold under a variety of names such as “Lye”. Sodium hydroxide is prepared on an industrial scale in a procedure called the “chloro-alkali” process. In the chloro-alkali process, a sodium chloride solution is electrolysized in a special cell composed of two compartments separated by a porous membrane. Chlorine gas is produced at the positive anode, and sodium hydroxide forms at cathode.

Process for the preparation of sodium hydroxide

Summary: Sodium hydroxide can be prepared by electrolyzing a sodium chloride solution in a two-compartment cell separated by a porous membrane. Chlorine gas is liberated at the positive anode and hydrogen and sodium hydroxide are liberated at the cathode. *Use proper ventilation when running the electrolysis cell because of chlorine and hydrogen gas evolution. Run the cell in an area that is away from direct sunlight.*

Hazards: Chlorine gas is produced in this procedure; either properly vent the gas, or neutralize it by bubbling it through a sodium hydroxide or sodium carbonate solution. Carryout this procedure away from direct sun-light, and keep any source of ignition away; hydrogen gas is very flammable and explosive.

Procedure: Prepare a cell as shown in the following illustration, and then add 500 grams of table salt (sodium chloride preferable sold under the name "pickling salt") to a beaker and then add 1500 milliliters of water. Then stir the mixture to dissolve the table salt. After which, pour 1000 milliliters of water into the apparatus anode compartment. Then pour about 100 milliliters of the sodium chloride solution into the anode compartment to bring its total volume to about 1100 milliliters. Afterwards, pour the rest of the sodium chloride solution into the apparatus cathode compartment. Then put the graphite electrodes in place and electrolysis at 12-amp/12-volt until no more chlorine gas is evolved. When no more chlorine is evolved, stop the electrolysis. Then pour the cathode liquid into a beaker, and then filter to remove any insoluble materials. After filtering, pour the sodium hydroxide solution into a clean stainless steel beaker and then boil-off the water until dry sodium hydroxide solid remains. Do not use glass when boiling-off the water because the sodium hydroxide will corrode the glass and cause it to break.



Apparatus for the production of sodium hydroxide. Chlorine gas is liberated at the positive anode electrode, and hydrogen gas is liberated at the negative cathode electrode. The sodium hydroxide is formed at the negative cathode electrode and remains dissolved in water.

Sodium nitrate

$NaNO_3$

Sodium nitrate forms colorless transparent crystals, or white powder or granules. The salt has a melting point of 308 Celsius, and is readily soluble in water with lowering of temperature; however sodium nitrate is relatively insoluble in most solvents. Sodium nitrate is a moderate oxidizer that forms explosive mixtures with combustible materials. It is a major by-product in the manufacture of nitric acid, and can be made in the lab by dissolving sodium carbonate into dilute nitric acid followed by recrystallization; however, sodium nitrate is so easily obtainable from numerous sources at very inexpensive prices.

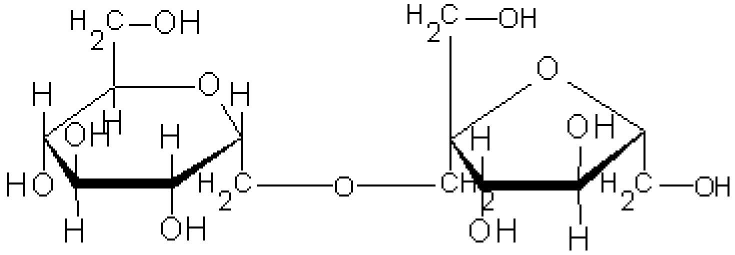
Sodium sulfate

Na_2SO_4

Sodium sulfate

Sodium sulfate occurs in nature as the minerals *mirabilite,* and *thenardite*. The anhydrous salt is called salt cake, and it forms a white powder or orthorhombic bipyramidal crystals. It has a melting point of 800 Celsius, and is moderately soluble in water. Sodium sulfate is insoluble in alcohol. The decahydrate is called glauber's salt, and it forms odorless, efflorescent crystals or granules, with a melting point of 32 Celsius. It becomes anhydrous when heated at 100 Celsius for 1 hour. It is soluble in water, and glycerol, but insoluble in alcohol. Sodium sulfate is a common salt of sulfuric acid, and can be made by reacting a water solution of Epsom salt with a water solution of sodium hydroxide or carbonate, and then filtering-off the precipitated by-product followed by recrystallizing the sodium sulfate from the water. The Sodium sulfate crystals are then heated to 100 Celsius for 1 to 2 hours to form the anhydrous salt. Sodium sulfate can also be made by neutralizing a dilute sulfuric acid solution with sodium hydroxide or carbonate, and then recrystallizing the sodium sulfate from the water. The sodium sulfate crystals are then heated to 100 Celsius for 1 to 2 hours to form the anhydrous salt. Sodium sulfate is a widely available commercial chemical.

Sucrose, granulated



Sucrose

Sucrose forms brilliant monoclinic spehnoidal crystals, granules, or white powder. The crystals show remarkable stability in air, and can be stored indefinitely. Sucrose begins to discolor and caramelize when heated to 160 Celsius. The crystal are very soluble in water, but relatively insoluble in alcohol and most common organic solvents. Sucrose is easily obtained from cane sugar or sugar beats by refining to yield high purity crystals. Sucrose is decomposed by dilute acids, bases, or enzymes forming invert sugar and containing a 50:50 mixture of glucose and fructose.

Sulfur

S

Sulfur

Sulfur forms a brilliant yellow solid with a melting point of 94 Celsius. It changes to the monoclinic form at 94 Celsius, which has a melting point of 115 Celsius. The monoclinic form changes back to the alpha form below 94 Celsius. Sulfur is insoluble in water, but soluble in carbon disulfide. It is only slightly soluble in benzene, acetone, and methylene iodide. It burns to sulfur dioxide when ignited, and combines with chlorine and bromine at room temperature. It combines with nearly all metals when both are in finely divided form, and heated. Sulfur is readily available commercially, and can be found in plant and garden stores.

Sulfur monochloride (also known as sulfer dichloride)

S_2Cl_2

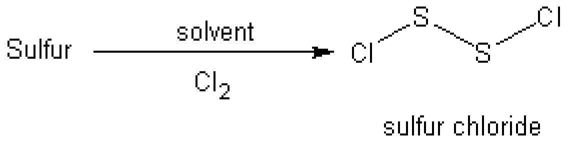
Sulfur monochloride forms a non-flammable, light amber to yellowish red, fuming oily liquid, which has a penetrating odor. It has a melting point of –77 Celsius, and a boiling point of 138 Celsius. It is soluble in alcohol, ether, carbon disulfide, toluene, carbon tetrachloride, and many oils. It reacts with water yielding sulfur dioxide, hydrogen chloride, and other products. Over exposure may cause skin irritation. It is conveniently prepared by passing dry chlorine into molten sulfur, and then recovering the sulfur chloride by distillation.

Method 1: Preparation of Sulfur monochloride from sulfur and chlorine (as seen in The Preparatory Manual of Explosives by Jared Ledgard)

Materials:

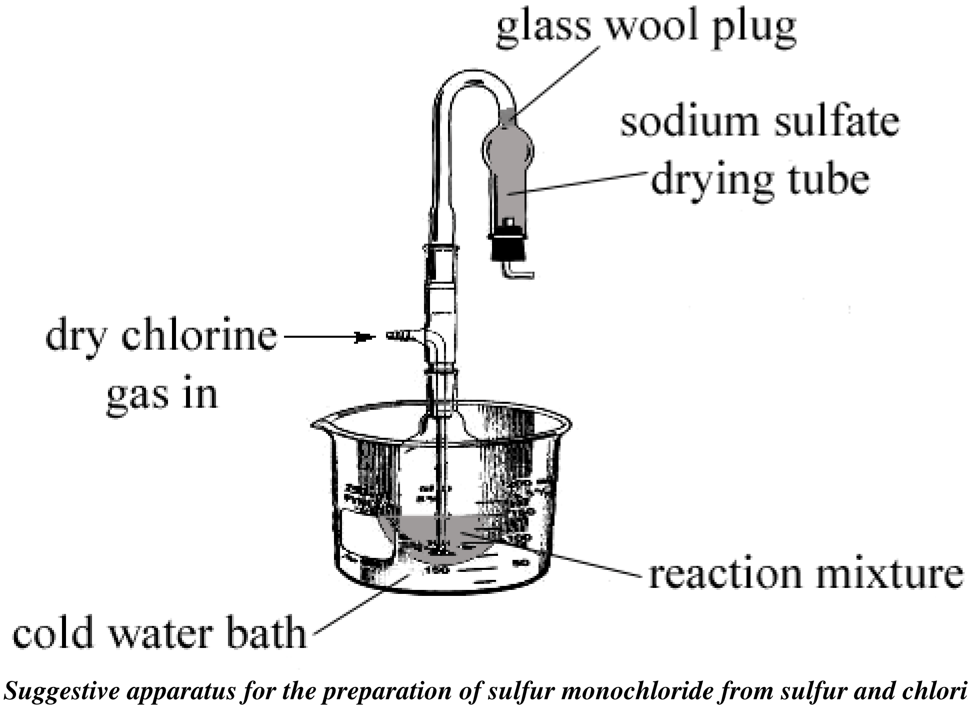
1. 300 milliliters (10 fluid oz.) of dry methylene chloride, or benzene	3. 26 grams (0.9 oz.) of dry chlorine gas
2. 24 grams (0.8 oz.) of powdered sulfur	

Reaction summary: Sulfur monochloride is readily prepared by reacting dry chlorine gas with powdered sulfur. The reaction is carried out in an inert solvent such as methylene chloride or benzene. After the reaction is complete, the reaction mixture is quickly filtered, and then distilled to remove the methylene chloride or benzene solvent.



Hazards: Use caution when handling chlorine gas, and use proper ventilation.

Procedure: Into an appropriate flask (as illustrated in the following illustration), place 300 milliliters (10 fluid oz.) of dry methylene chloride, or benzene, and then add in 24 grams (0.8 oz.) of powdered sulfur. Thereafter, place the flask into a cold-water bath, and then pass 26 grams (0.9 oz.) of dry chlorine gas into the mixture over a several hour period while rapidly stirring the reaction mixture. During the addition, keep the reaction mixture below 30 Celsius. After the addition of the chlorine gas, quickly filter the reaction mixture to remove any insoluble materials, and then place the filtered reaction mixture into a distillation apparatus (not illustrated below; the distillation apparatus can be any simple apparatus), and then boil-off the methylene chloride at 40 Celsius, or if using benzene, boil at 80 Celsius to drive-off the benzene. When no more methylene chloride or benzene distills over, remove the heat source, and then allow the remaining oily liquid of sulfur monochloride to cool to room temperature. Then pour the remaining oily liquid into any suitable dry amber glass bottle, and store in a cool dry place.



Suggestive apparatus for the preparation of sulfur monochloride from sulfur and chlorine.

Sulfur dioxide

$$SO_2$$

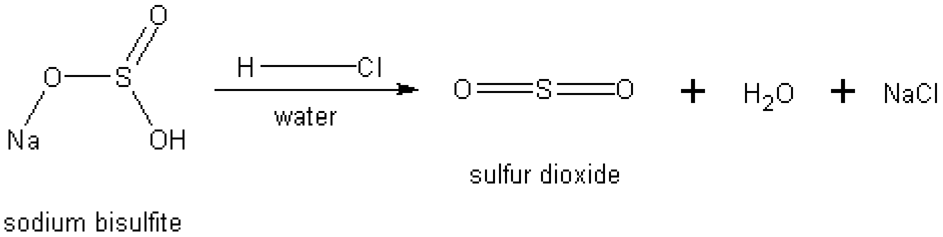
Sulfur dioxide forms a colorless gas with a strong suffocating and irritating odor. The gas has a melting point of –72 Celsius, and a boiling point of –10 Celsius. The gas is easily condensed into a colorless liquid. The gas is soluble in water, alcohol, chloroform, and ether. Sulfur dioxide is available in gas cylinders, but it can be prepared in the lab by dripping hydrochloric acid onto excess sodium bisulfite.

Method 1: Preparation of sulfur dioxide gas from sodium bisulfite and hydrochloric acid

Materials:

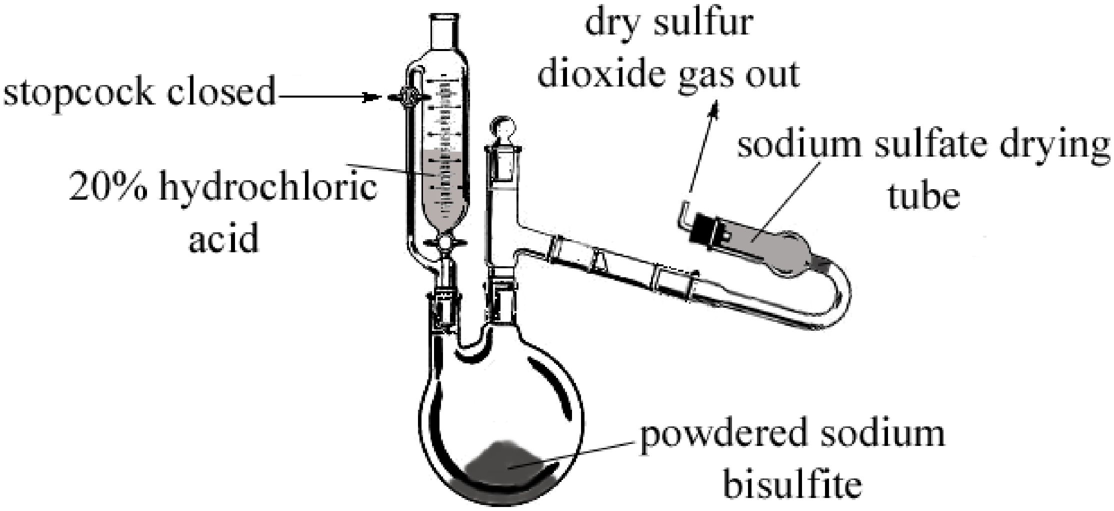
1. 100 grams (3.5 oz.) of powdered sodium bisulfite	2. 100 grams (3.5 oz.) of concentrated hydrochloric acid

Reaction summary: Sulfur dioxide gas is easily prepared by mixing hydrochloric acid with sodium bisulfite. The reaction is very smooth and generates a steady stream of gas.



Hazards: Use proper ventilation when using sulfur dioxide gas, which is very irritating to the nose and throat. Avoid inhalation of the fumes.

Procedure: Set-up the apparatus as illustrated below, and then place 100 grams (3.5 oz.) of powdered sodium bisulfite into the flask as illustrated, and then place 100 grams (3.5 oz.) of concentrated hydrochloric acid or concentrated Muriatic acid into the addition funnel as illustrated. Thereafter, gradually drip the hydrochloric acid onto the sodium bisulfite over a period of about 30 to 90 minutes. During the acid addition, the sulfur dioxide gas will be steadily evolved and will carry over.


stopcock closed
dry sulfur dioxide gas out
sodium sulfate drying tube
20% hydrochloric acid
powdered sodium bisulfite

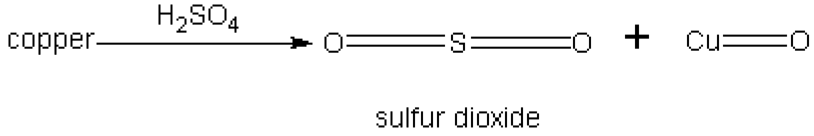
Set-up for the generation of sulfur dioxide gas.

Method 2A: Preparation of sulfur dioxide gas from sulfuric acid and copper

Materials:

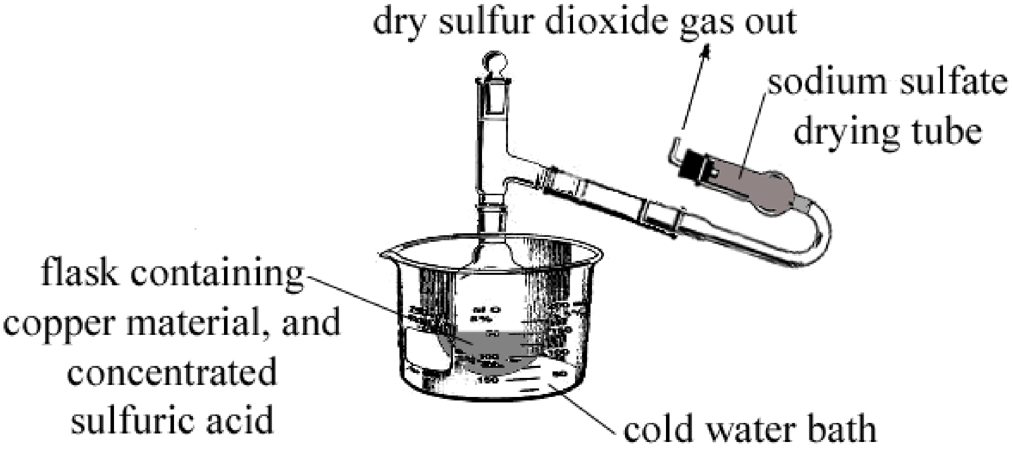
1. 150 grams (5.2 oz.) of concentrated sulfuric acid	2. 97 grams (3.4 oz.) of copper wire, pipes, pieces, or any other pure copper material

Reaction summary: Sulfur dioxide gas is readily prepared by reacting concentrated sulfuric acid with copper. The reaction is smooth and produces a steady stream of gas.



Hazards: Use proper ventilation when using sulfur dioxide gas, and avoid inhalation of the fumes. Wear gloves when handling concentrated sulfuric acid, and use caution.

Procedure: Into the apparatus illustrated below, place 150 grams (5.2 oz.) of concentrated sulfuric acid, followed by 97 grams (3.4 grams) of copper wire, pipes, pieces, or any other pure copper material, and then seal the apparatus using a glass stopper as illustrated below. Thereafter, monitor the reaction and do not let the acid mixture get to hot. During the reaction, a steady stream of sulfur dioxide gas will be steadily evolved. The total reaction time can be anywhere from 45 minutes to 2 hours. Heat may or may not be applied to speed up the reaction.


dry sulfur dioxide gas out
sodium sulfate drying tube
flask containing copper material, and concentrated sulfuric acid
cold water bath

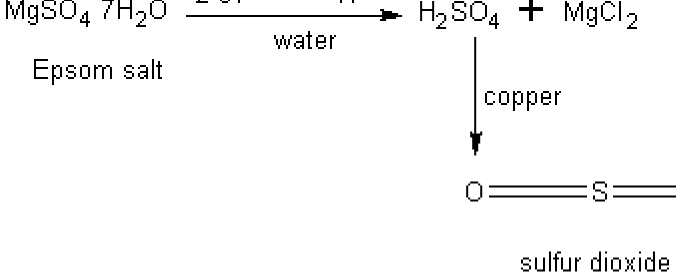
Set-up for the preparation of sulfur dioxide gas from sulfuric acid and copper.

Method 2B: Preparation of sulfur dioxide gas from Epsom salt, hydrochloric acid, and copper

Materials:

1. 400 grams (14.1 oz.) of Epsom salt	3. 330 grams (11.6 oz.) of concerted hydrochloric acid or concentrated Muriatic acid
2. 1300 milliliters (1.3 quarts) of warm water	4. 103 grams (3.6 oz.) of copper wire, pipes, pieces, or any other pure copper material

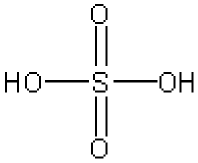
Reaction summary: Sulfur dioxide gas can be readily prepared in a modified process using Epsom salt rather then sulfuric acid. In this regards, the concentrated hydrochloric acid partially reacts with the Epsom salt forming the acidic bisulfate ion, and sulfuric acid. These substances then in turn, react with copper to produce sulfur dioxide and the usual by-products.



Hazards: Use proper ventilation when using sulfur dioxide gas, and avoid inhalation of the fumes. Wear gloves when handling concentrated hydrochloric acid, and avoid inhalation of the fumes.

Procedure: Into a large beaker or similar container, place 400 grams (14.1 oz.) of Epsom salt, followed by 1300 milliliters (1.3 quarts) of warm water, and then stir the mixture briefly to dissolve all of the Epsom salt. Thereafter, pour this Epsom salt solution into a similar apparatus as illustrated in method 2A, and then add in 330 grams (11.6 oz.) of concentrated hydrochloric acid or concentrated Muriatic acid, and then blend the mixture for about 10 minutes to form a uniform acidic mixture. Note: an exothermic reaction will proceed, lowering the temperature of the acidic solution. Thereafter add in 103 grams (3.6 oz.) of copper wire, pipes, pieces, or any other pure copper material, and then seal the apparatus using a glass stopper. Thereafter, monitor the reaction and do not let the acid mixture get to hot. During the reaction, a steady stream of sulfur dioxide gas will be steadily, yet slowly evolved. The total reaction time can be anywhere from 2 hours to 4 hours. Heat may or may not be applied to speed up the reaction.

98% Sulfuric acid



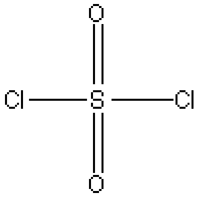
Sulfuric acid

98% Sulfuric acid is a clear, colorless, odorless, oily liquid, which is very corrosive. 98% Sulfuric acid is referred to as concentrated sulfuric acid. Some grades of concentrated sulfuric acid may have a slight amber to brown tint due to ferrous sulfate impurity. Concentrated sulfuric acid has a great affinity for water; and hence, will char or dehydrate a great many substances. It chars wood, fabrics, resins, and also dehydrates sugar, forming carbon. Concentrated sulfuric acid has a boiling point of 290 Celsius. It decomposes when heated to 340 Celsius producing sulfur trioxide fumes and water. It has a melting point of 10 Celsius. Large amounts of heat are produced when concentrated sulfuric acid mixes with water or alcohol. When mixing with water, the acid should slowly be added. Never add the water to the concentrated acid. Concentrated sulfuric acid is a widely available commercial acid. It is the largest manufactured chemical in the world. Concentrated sulfuric acid is prepared on an industrial scale from sulfur dioxide by oxidation of sulfur or sulfides. Afterwards, the sulfur dioxide is converted into sulfur trioxide by oxidation with air over a platinum or vanadium pentoxide catalyst at 500 Celsius. The sulfur trioxide is then absorbed into 98% sulfuric acid forming 100% fuming sulfuric. This in turn is then treated with the calculated amount of water to form two parts of 98% sulfuric acid. The first part is then recycled for further absorption, and the second part is bottled and shipped. *Wear gloves when handling concentrated sulfuric acid. Concentrated sulfuric acid is a very corrosive and toxic liquid. It can cause severe skin burns and irritation. Wear proper protective clothing (certified lab coat) when handling sulfuric acid because acid spills on cloths can cause a "melting" effect of the fabric. Note: In one documented case a laboratory student working in the lab, spilled concentrated sulfuric acid onto her nylons causing the nylon fibers to "melt" to her skin. She had to be rushed to the emergency room where doctors had to surgically remove each nylon fiber.* **Note:** recovering sulfuric acid from water solutions can be accomplished by heating the sulfuric acid/water solution to 110 Celsius, and heating until no more water is evolved. This produces 93 to 98% sulfuric acid, which is perfectly suitable for use as 98% sulfuric acid (to determine when no more water can be evolved, place a piece of glass over the heated mixture. If the glass fogs-up, water is still being evolved from the sulfuric acid). **Hazards:** monitor the heating process very closely. Thick walled laboratory glass vessels should be avoided. Watch for white fumes, and be aware of potential irritating vapors; some decomposition of the acid may result.

20% sulfuric acid solution

Prepare by carefully dissolving 150 grams of 98% sulfuric acid into 600 milliliters of water.

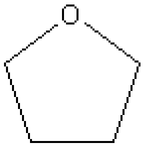
Sulfuryl chloride



Sulfuryl chloride

Sulfuryl chloride forms a mobile, colorless liquid, which has an unpleasant pungent odor. The liquid and its vapors are corrosive to skin, tissue, hair, and mucous membranes. The liquid has a melting point of –54 Celsius, and a boiling point of 69 Celsius. It slowly turns yellow on standing, and decomposes slowly in water forming sulfuric acid and hydrogen chloride. It is made by mixing dry sulfur dioxide gas with dry hydrogen chloride gas, usually in the presence of activated charcoal. Sulfuryl chloride reacts violently on contact with alkalis.

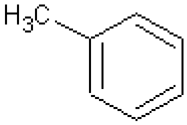
Tetrahydrofuran. *Diethylene oxide; Tetramethylene oxide*



Tetrahydrofuran

Tetrahydrofuran (THF) is a colorless liquid, with an ether-like odor. It has a melting point of –108 Celsius, and a boiling point of 66 Celsius. It is miscible with water, alcohols, acetone, ethyl acetate, ether, and hydrocarbon solvents. Tetrahydrofuran slowly forms peroxides if stored in the presence of air. For storage, fill the bottle as full as possible (leaving as little air-gap as possible). Before distilling teteahydrofuran or its mixtures, always add ferrous sulfate to check for, and destroy peroxides (a red or black color will appear). Explosions will result if peroxide contaminated tetrahydrofuran is distilled without treatment with ferrous sulfate, or other reducing agents. Tetrahydrofuran is a common solvent, and occurs in many commercial products. One source of tetrahydrofuran is PVC cement, which can be distilled to recover tetrahydrofuran. Tetrahydrofuran is a commercially available solvent.

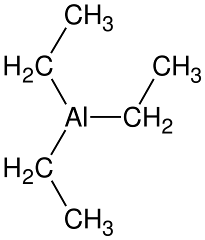
Toluene



Toluene

Toluene is a flammable, refractive liquid with a benzene like odor. It has a melting point of –95 Celsius, and a boiling point of 110 Celsius. Toluene is insoluble in water, but miscible with alcohol, chloroform, ether, acetone, glacial acetic acid, and carbon disulfide. Toluene is toxic. It can be obtained from tar oil, and is a very common commercial solvent.

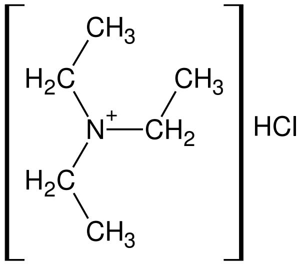
Triethyl Aluminum



triethyl aluminum

Triethyl aluminum is a colorless liquid, that is very dangerous as it ignites spontaneously in air burning with a white hot flame. It is made by reacting 3 parts ethyl chloride with 1 part of powdered aluminum in an inert solvent, followed by distillation in the absence of air.

Triethyl amine hydrochloride



triethylamine hydrochloride

Triethylamine hydrochloride form white crystalline solid, powder, or granules, with a melting point of 253 Celsius, but begins to sublime at 245 Celsius. The compound is soluble in water, chloroform, but very slightly soluble benzene, and insoluble in ether. It is prepared by first reacting ethanol with ammonia on an industrial scale using a catalyst, followed by dissolving the triethylamine into dilute hydrochloric acid followed by recrystallization.

Vanadium pentoxide

$$V_2O_5$$

Vanadium pentoxide forms yellow to rust brown orthorhombic crystals with a melting point of 690 Celsius. Begins to lose oxygen when heated to above 700 Celsius+. Vanadium pentoxide is a powerful catalyst, and converts sulfur dioxide to sulfur trioxide. The compound is made by heating ammonium metavanadate.

Xenon gas

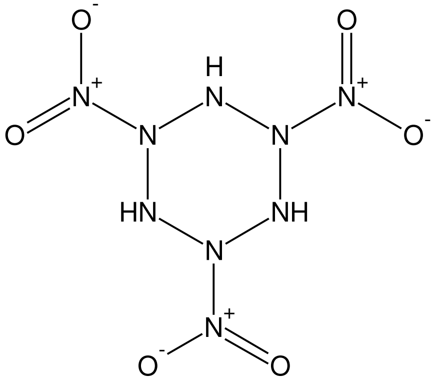
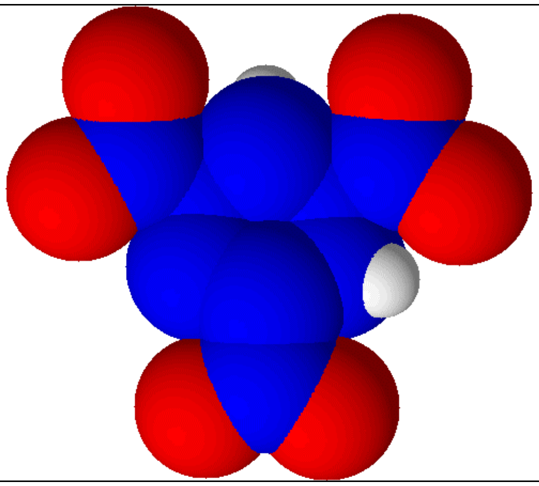
$$Xe$$

Xenon is a colorless, odorless, tasteless gas, that is considered a noble gas, but it does form compounds readily with fluorine under the proper conditions. Xenon is quite rare, and can be quite expensive.

Section 3: Experimental Explosives Chemistry

Note: all detonating velocities are given in meters/per/second.

Theoretical Preparation 1: 1,3,5-trinitrohexazinane; Azinane

 1,3,5-trinitrohexazinane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_3N_9O_6$
Formula Weight = 225.08052
Composition = H(1.34%) N(56.01%) O(42.65%)
Molar Refractivity = 40.71 ± 0.4 cm^3
Molar Volume = 91.0 ± 5.0 cm^3
Parachor = 340.5 ± 6.0 cm^3
Index of Refraction = 1.851 ± 0.03
Surface Tension = 195.9 ± 5.0 dyne/cm
Density = 2.47 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 16.14 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 225.020629 Da
Nominal Mass = 225 Da
Average Mass = 225.0805 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
10,978 (world record).	Burns with colorless flame emitting red fumes.	Stable at room temperature, and temperatures up to 180 Celsius; Stable towards shock,	Reacts with strong bases forming highly sensitive salts that easily detonate; keep away from strong	A secondary explosive. Most likely forms a colorless to light reddish crystalline

		percussion, pressure, and friction (requires blasting cap or detonator for initiation). A secondary explosive.	bases. Do not mix with metallic lithium, sodium or potassium as explosion may result. Moderately stable to oxidizers, but mixing with strong oxidizers like potassium permanganate, calcium hypochlorite, or potassium dichromate may form shock sensitive and friction sensitive mixtures that may ignite spontaneously.	mass forming cubic prisms. The melting point ranges from 123 to 156 Celsius depending on purity. The compound detonates when strongly and rapidly heated to above 200 Celsius. The compound is insoluble in water, but somewhat soluble in methylene chloride, carbon tetrachloride; good solubility in liquid ammonia.

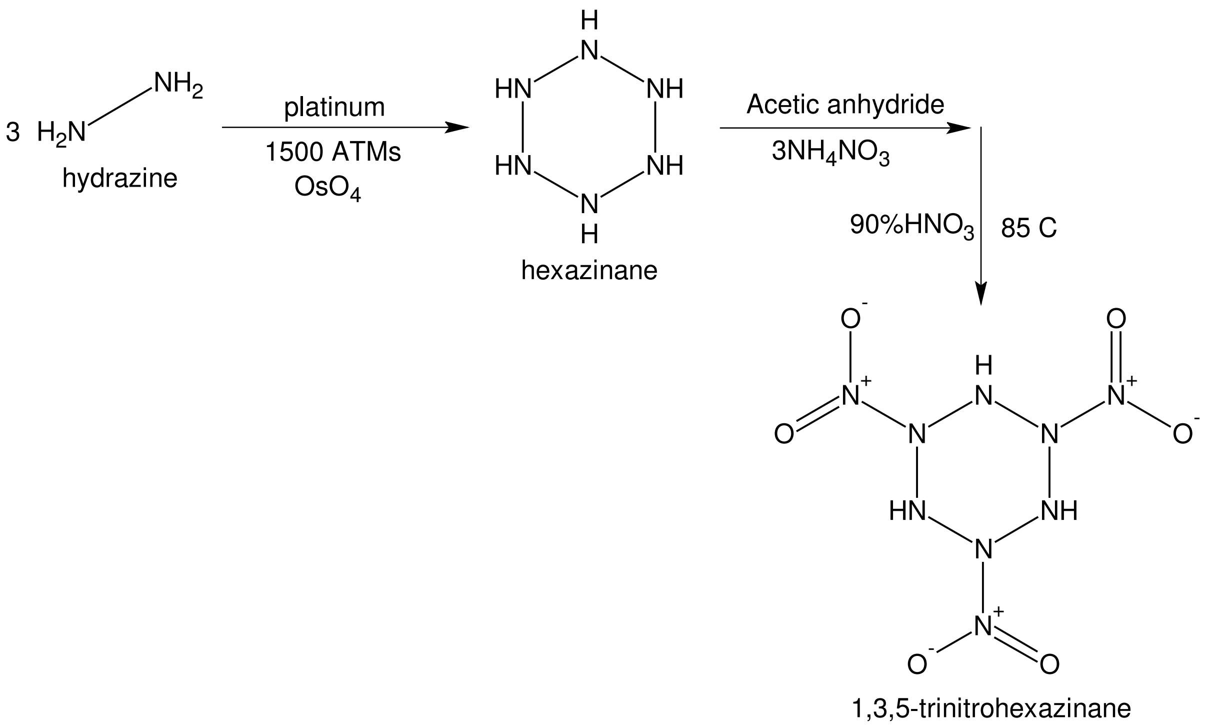
Estimated Compound summary: 1,3,5-trinitrohexazinane shows remarkable explosive power, and may be used primarily in drop bombs, laser guided bombs, cruise missile warheads, and other missile warheads. Due to its remarkable stability,1,3,5-trinitrohexazinane can either be pressed into bomb casings at 1500 psi, or melted down, and poured into molds for making a variety of explosive munitions such as shape charges for anti-tank weapons, or poured into bomb casings for mortar shells or artillery shells for a variety of explosive operations. Note: molten 1,3,5-trinitrohexazinane is insoluble in all known common explosives such as TNT, RDX, and HMX ect. 1,3,5-trinitrohexazinane can also be mixed with nitrocellulose or ammonium nitrate and an inert filler such as aluminum oxide for making blasting dynamites primarily for military use. One major attribute that this compound demonstrates is its ability to react with strong bases such as sodium or potassium hydroxides, to form shock sensitive salts that easily detonate when struck with a hammer, and touched by a spark or flame. The highly sensitive salts could be used as primary explosives in blasting caps and detonators when diluted with dextrose to a ratio of 3 parts explosive salt to 7 parts dextrose. The compound may also demonstrate a use in high performance rockets propellants, as a single entity or the 1,3,5-trinitrohexazinane can be mixed with reducing agents mainly powdered aluminum, and then cured with epoxy for making high performance rocket fuels. 1,3,5-trinitrohexazinane should not be used in making gun propellants for small arms weapons as exploding gun chambers can result, but the explosive can be used to make gun propellants for large caliber guns such as field artillery and battleship guns—Note: these gun propellants have tremendous chamber pressures in excess of 230,000 psi so extreme caution should be taken, and proper testing should be conducted.

Safety Info: Wear gloves when handling osmium tetraoxide, as it is toxic. Acetic anhydride is corrosive so wear gloves when handling. Wear gloves and use proper ventilation when handling 90% nitric acid, which gives off reddish fumes, which are poisonous. Use caution when handling anhydrous hydrazine, never heat it or boil it in the presence of oxygen to avoid possible explosion. Keep anhydrous hydrazine away from reducing agents, and oxidizing agents to prevent possible violent explosions.

Materials:

1. Dry nitrogen gas from tank	6. 400 grams of ammonium nitrate
2. 1 gram of platinized asbestos	7. 190 milliliters of 90% nitric acid
3. 50 grams of Osmium tetraoxide	8. 1500 milliliters of ice water
4. 150 grams of pure dry anhydrous hydrazine	9. Six-500 milliliter portions of hot water
5. 250 milliliters of acetic anhydride	10. Three-500 milliliter portions of a 5% sodium carbonate solution

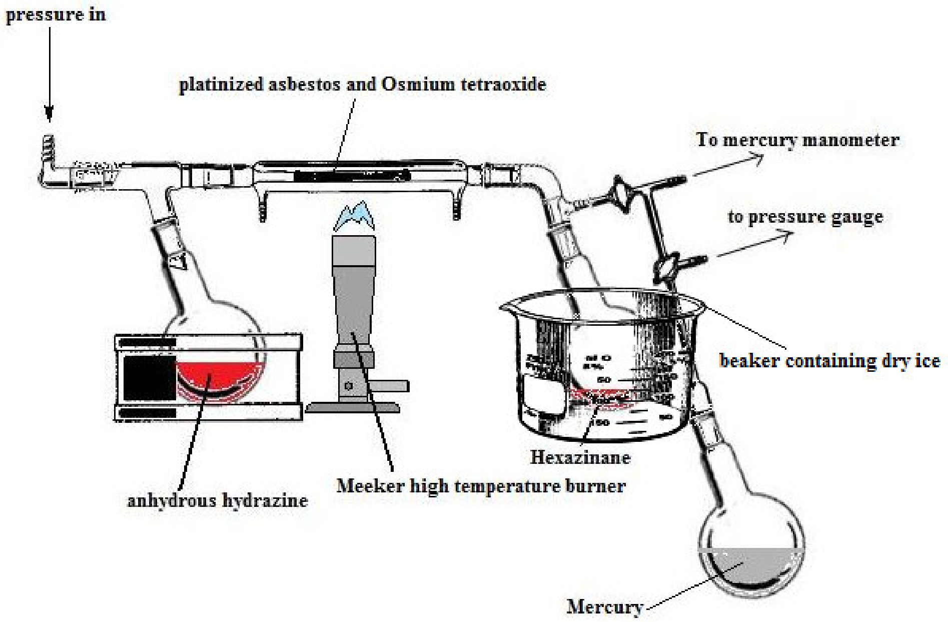
Reaction Equation:



Preparation:

Step 1: Preparation of Hexazinane

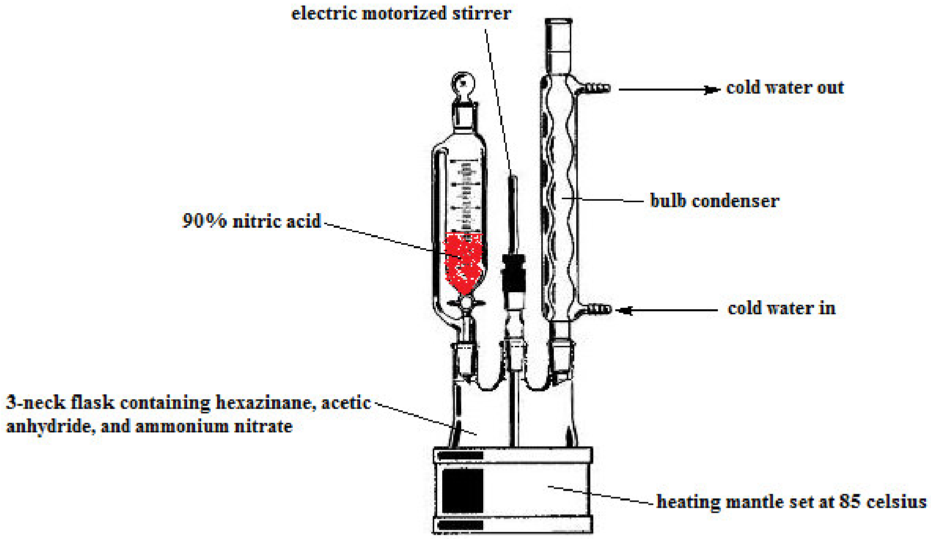
Note: prior to starting, flush the entire apparatus with dry nitrogen to expel any oxygen or carbon dioxide, and then maintain a pressure of 1500 ATM's under nitrogen. Into a suitable apparatus as illustrated below, place 1 gram of platinized asbestos, followed by 50 grams of Osmium tetraoxide. Thereafter assemble the apparatus as illustrated below, and then place 150 grams of pure dry anhydrous hydrazine into the boiling flask as shown. Heat the anhydrous hydrazine to 450 Celsius using the heating mantle. **Warning! Make sure the entire apparatus is free from air otherwise an explosion will happen. Also, make sure to use proper stainless steel apparatus as 1500 ATMS will crack and burst ordinary laboratory glassware.**


pressure in
platinized asbestos and Osmium tetraoxide
To mercury manometer
to pressure gauge
beaker containing dry ice
Hexazinane
Meeker high temperature burner
anhydrous hydrazine
Mercury

Setup for preparation of Hexazinane. Stainless steel apparatus.

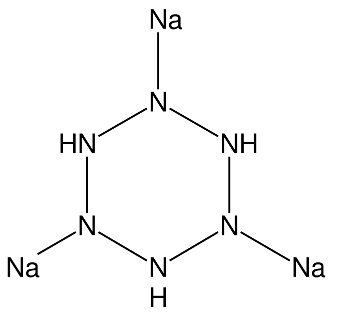
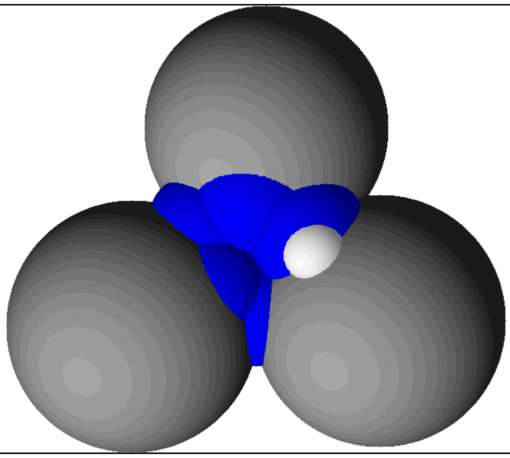
Step 2: Preparation of 1,3,5-trinitrohexazinane

Into an apparatus similar to the following illustrated below, place the hexazinane prepared in step 1 (let it thaw out back to a liquid before use) into the 3-neck flask, and then add in 250 milliliters of acetic anhydride, and then add in 400 grams of ammonium nitrate. Thereafter, begin to stir the mixture utilizing an electric stirrer as illustrated, and then immediately place 190 milliliters of 90% nitric acid into the dropping funnel. Then heat the mixture to 85 Celsius, and then drip in the 90% nitric acid drop by drop over a period of one hour while maintaining the reaction mixtures temperature around 85 Celsius with heavy stirring. After the addition of the 90% nitric acid, reduce heat of the heating mantle to 50 Celsius, and then continue to stir the reaction mixture for 4 hours at 50 Celsius. After 4 hours, turn off the heat, and allow the reaction mixture to come to room temperature; thereafter, pour the entire reaction mixture into 1500 milliliters of ice water, and then stir for about 15 minutes. After 15 minutes, filter-off the insoluble mass, and then wash (using vacuum suction) with three-500 milliliter portions of hot water, followed by three-500 milliliter portions of a 5% sodium carbonate solution, and then finally followed by another three-500 milliliter portions of warm water, and then suction dry the mass as best a possible. Thereafter, place the filter cake into an oven and heat at 105 Celsius for 12 hours. After 12 hours, turn off the oven, and then allow the dried desired product to cool to room temperature. Once it has cooled, remove it from the oven, and then store it in a glass container in a cool dry place until use.


electric motorized stirrer
cold water out
bulb condenser
90% nitric acid
cold water in
3-neck flask containing hexazinane, acetic anhydride, and ammonium nitrate
heating mantle set at 85 celsius

Setup for preparation of 1,3,5-trinitrohexazinane.

Theoretical Preparation 2: trisodium hexazinane-1,3,5-triide; SOD

 trisodium hexazinane-1,3,5-triide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_3N_6Na_3$

Formula Weight = 156.033327

Composition = H(1.94%) N(53.86%) Na(44.20%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 156.011229 Da
Nominal Mass = 156 Da
Average Mass = 156.0333 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
Unknown; 2300 to 3400 estimated	Flammable solid. Burns violently producing white fumes of free sodium that spontaneously reacts with air forming the oxide. Warning! May react violently with water, leading to explosion.	Sensitive to shock, friction, and percussion. Easily detonated. A primary explosive.	Stable at room temperature when dry, but hydrolysizes in moist air, and may explode in contact with water.	Colorless, crystalline powder, or granules. Melting point unknown; boiling point unknown. The compound is hydrolysized in water, and has no solubility in hexane, toluene, benzene, or methylene chloride, but has moderate solubility in mercury.

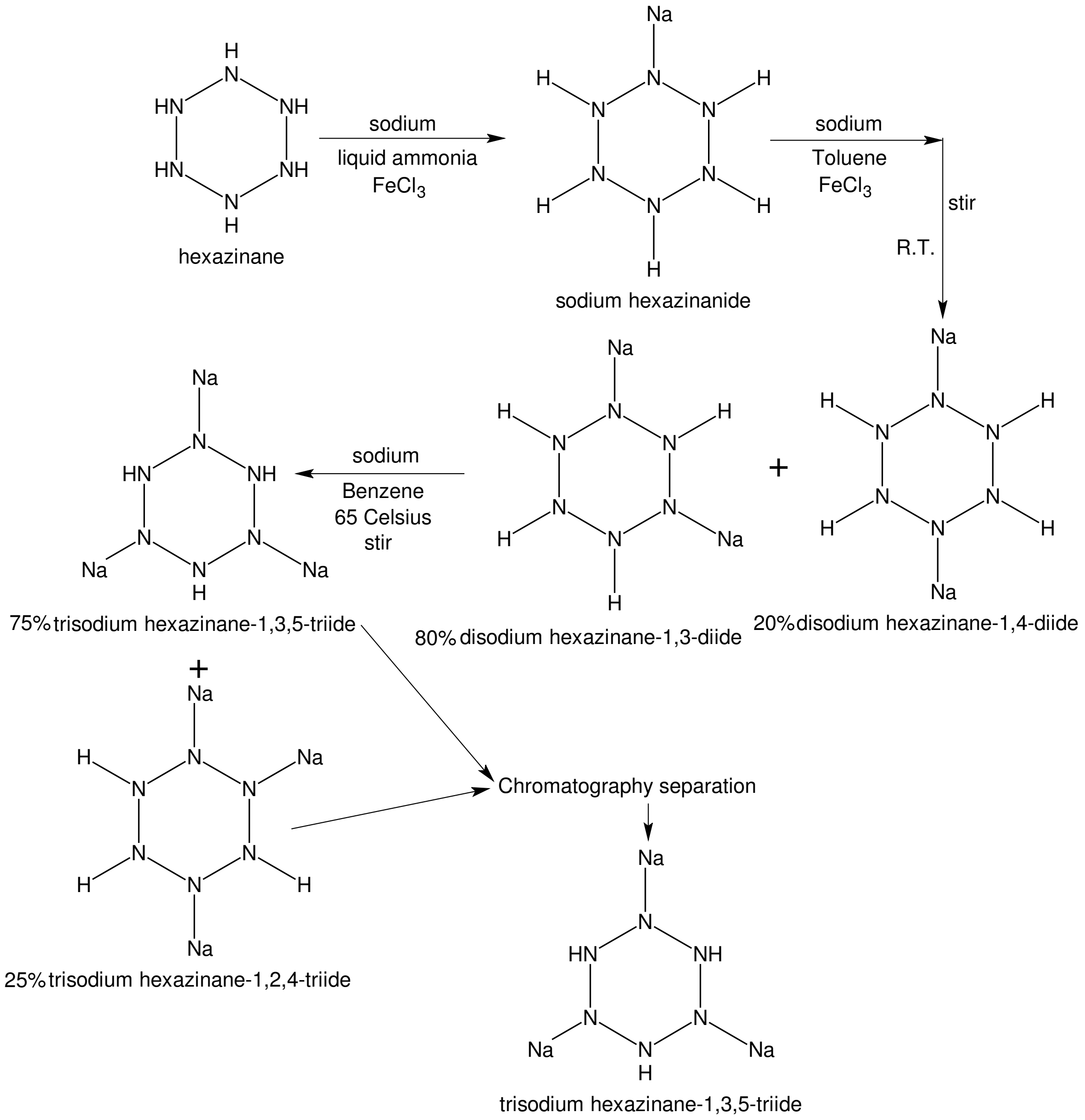
Estimated Compound summary: trisodium hexazinane-1,3,5-triide, is a theoretical primary explosive designed to become a substitute for lead azide, and the out dated mercury fulminate for use in blasting caps, and detonators. Even though the compound is sensitive to shock, it does have enough stability to be used in blasting caps and detonators for use in high velocity explosive munitions such as tank fired projectiles, and artillery shells, and it shows remarkable stability in this field. trisodium hexazinane-1,3,5-triide shows potential for use in fireworks, for aerial displays, as it explodes with great brilliance when mixed with powered metals such as aluminum, magnesium, iron, or zinc. Also, trisodium hexazinane-1,3,5-triide could be a replacement for the old lead styphnate in blasting caps and detonators. Note: The only drawback to this substance is its difficulty in manufacture and cost. **Note:** Even though hexazinane reacts with strong bases forming shock sensitive salts, the reactions are very non-descriptive, and often lead to rather unorganized products with very difficulty in isolating and purifying a single desired component, and water is often the by-product and reacts with the mixture of shock sensitive salts hydrolyzing them, so in order to form our desired product here, we must use this procedure in order to achieve the desired compound!

Safety Info: Wear gloves and respiration mask and use proper ventilation when handling liquid ammonia. Do not touch with bare hands. Use severe caution when handling metallic sodium and wear gloves when handling, use extreme caution as metallic sodium is highly reactive, and explodes in contact with many substances especially water. Use proper ventilation, and avoid inhalation of fumes when handling toluene and benzene, which are carcinogens. Extinguish all flames when handling acetone as it is highly flammable. Wear gloves and avoid skin contact when handling phosphorus pentoxide, which is a strong dehydrating agent. Wear gloves when handling potassium hydroxide, which is highly corrosive.

Materials:

1. Dry nitrogen gas from tank	11. 150 grams of phosphorus pentoxide
2. 150 milliliters of liquid ammonia	12. 100 grams of fine coarse sand
3. 10 grams of anhydrous ferric chloride	13. 100 grams of boric anhydride
4. 124 grams of hexazinane (prepared in theoretical preparation 1, step 1)	14. 150 grams of tetrapotassium EDTA
5. 96 grams of metallic sodium	15. 100 grams of lauryl sulfate
6. 500 milliliters of Toluene	16. 100 grams of triethyl amine hydrochloride
7. 1200 milliliters of benzene	17. 200 grams of aluminum oxide
8. 500 milliliters of methylene chloride	18. 96 grams of potassium hydroxide
9. 250 milliliters of a 30% ferric chloride hexahydrate solution	19. 750 milliliters of 99% hexane
10. 500 milliliters of acetone	

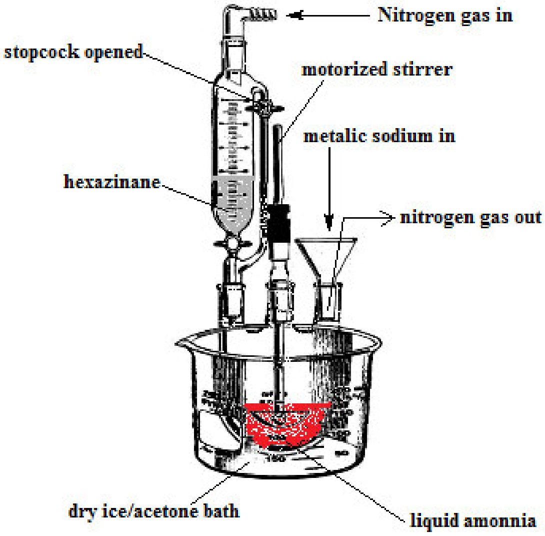
Reaction Equation:


hexazinane
sodium
liquid ammonia
FeCl3
sodium hexazinanide
sodium
Toluene
FeCl3
stir
R.T.
80% disodium hexazinane-1,3-diide
+
20% disodium hexazinane-1,4-diide
sodium
Benzene
65 Celsius
stir
75% trisodium hexazinane-1,3,5-triide
+
25% trisodium hexazinane-1,2,4-triide
Chromatography separation
trisodium hexazinane-1,3,5-triide

Preparation:

Step 1: Preparation of sodium hexazinanide

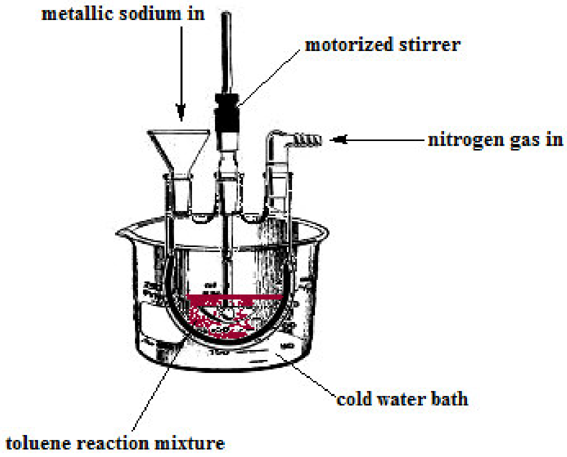
Into a suitable apparatus as illustrated below, place 150 milliliters of liquid ammonia, followed by 5 grams of anhydrous ferric chloride. Thereafter, begin to stir the mixture and then into one of the addition funnels place 124 grams of hexazinane, and then begin a slow drip of the hexazinane into the liquid ammonia; when the hexazinane begins to slowly drip into the liquid ammonia, add in very small portions (about 500 mg per portions) of metallic sodium, while rapidly stirring the liquid ammonia reaction mixture and maintaining its low temperature using a dry ice/acetone bath. Continue to add the metallic sodium in small portions until you have added a total of 32 grams of metallic sodium. After the addition of the metallic sodium, disassemble the apparatus, and allow the remaining liquid ammonia to evaporate (use a fume hood with good ventilation). After the rest of the ammonia has evaporated, remove the left over residue in the reaction flask, and the place this residue into 500 milliliters of Toluene, then stir for 30 minutes. Thereafter, filter the toluene mixture to remove any insoluble impurities (mostly sodium amide), and then keep the filtered toluene mixture for step 2. **Warning! Metallic sodium is very reactive, and will explode in contact with water.**



Apparatus for preparation of sodium hexazinanide.

Step 2: Preparation of disodium hexazinane-1,3-diide, and disodium hexazinane-1,4-diide

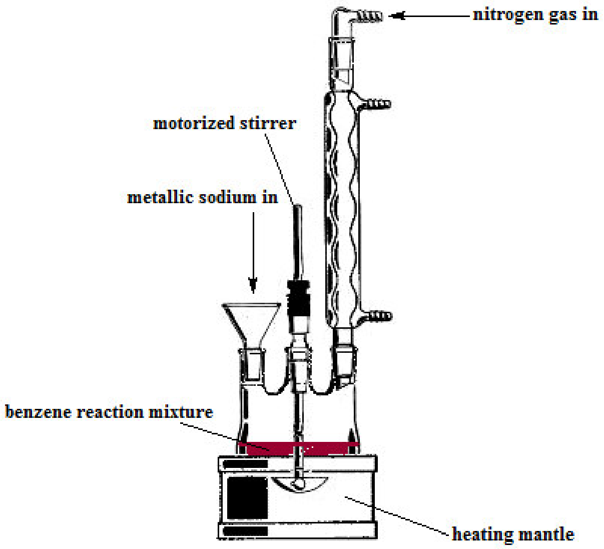
Into a suitable apparatus as illustrated below, place the filtered toluene mixture obtained at the end of step 1, and then add in 5 grams of anhydrous ferric chloride. Thereafter, begin to stir the toluene mixture, and then slowly, in small portions at a time (500 mg portions) add in metallic sodium while stirring the reaction mixture and maintaining its temperature below 30 Celsius at all times. Continue to stir and add in small portions of metallic sodium until a total of 32 grams of sodium has been added. After the addition continue to stir the reaction mixture at room temperature for about 1 hour. After 1 hour, quickly vacuum filter the reaction mixture to remove any insoluble impurities, and then place the filtered reaction mixture into a vacuum distillation apparatus, preferably a rotary evaporator, and distill off the toluene under high vacuum and under a temperature of 65 Celsius, until dry solid remains. When dry solid remains, stop the evaporation process, and then remove the dry residue and keep for step 3. **Warning! Metallic sodium is very reactive, and will explode in contact with water.**



Setup for preparation of disodium hexazinane-1,3-diide, and disodium hexazinane-1,4-diide.

Step 3: Preparation of trisodium hexazinane-1,3,5-triide, and trisodium hexazinane-1,2,4-triide

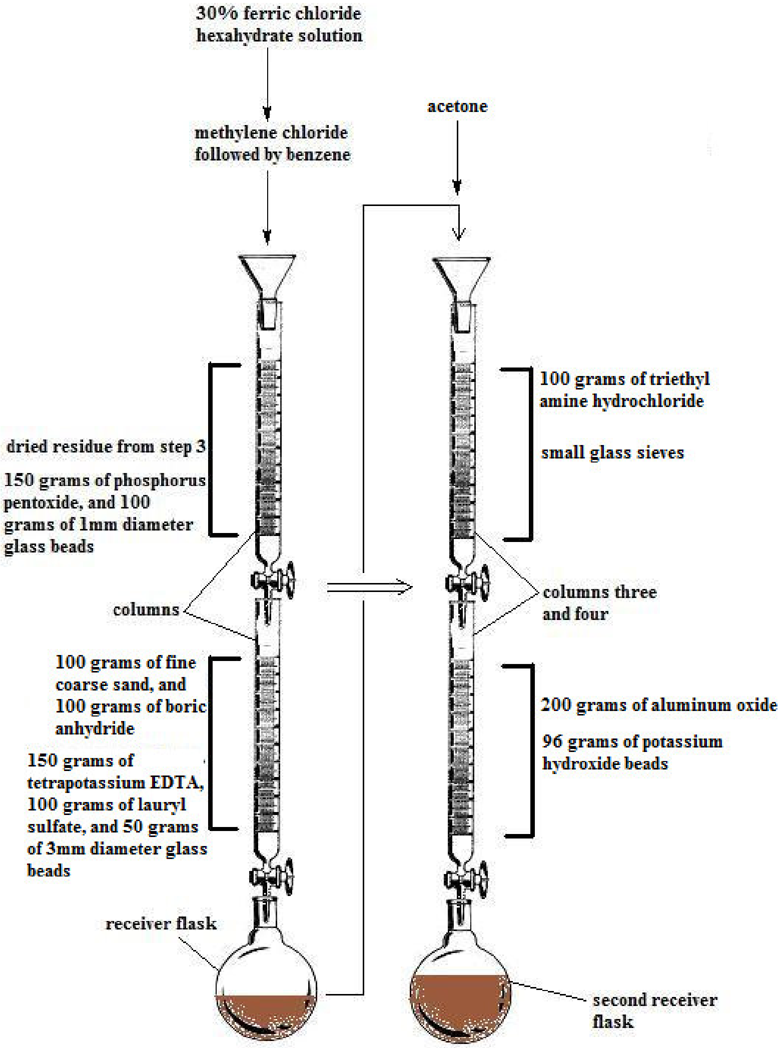
Into a suitable apparatus as illustrated below, place the dry residue from step 2, and then pour in 700 milliliters of benzene. Thereafter, begin to stir the mixture on medium speed, and then begin a gentle heating of the mixture until a temperature of 65 Celsius is achieved. When the temperature of benzene mixture reaches 65 Celsius, slowly add in small portions at a time (500 mg per portions) of metallic sodium while vigorously stirring the reaction mixture and maintaining its temperature below 70 Celsius. Note: Keep a close eye on the temperature, and do not allow the reaction mixture to go above 70 Celsius (turning the heating mantle off and on may need to be done in order keep the reaction mixtures temperature around 65 Celsius. Continue to add the metallic sodium in small portions until a total of 32 grams of metallic sodium has been added. After the addition of the metallic sodium, continue to rapidly stir the mixture and maintain a gentle heat around 65 Celsius for about 1 hour. After 1 hour, remove the heating source and allow the reaction mixture to come to room temperature. Then, filter the cooled reaction mixture to remove any insoluble impurities, and then place the filtered reaction mixture into a rotary evaporator, and remove the benzene under high vacuum and under a temperature of 70 Celsius until dry solid remains. When dry solid remains remove the left over dried residue and save for step 4. **Warning! Metallic sodium is very reactive, and will explode in contact with water.**



Setup for preparation of trisodium hexazinane-1,3,5-triide, and trisodium hexazinane-1,2,4-triide.

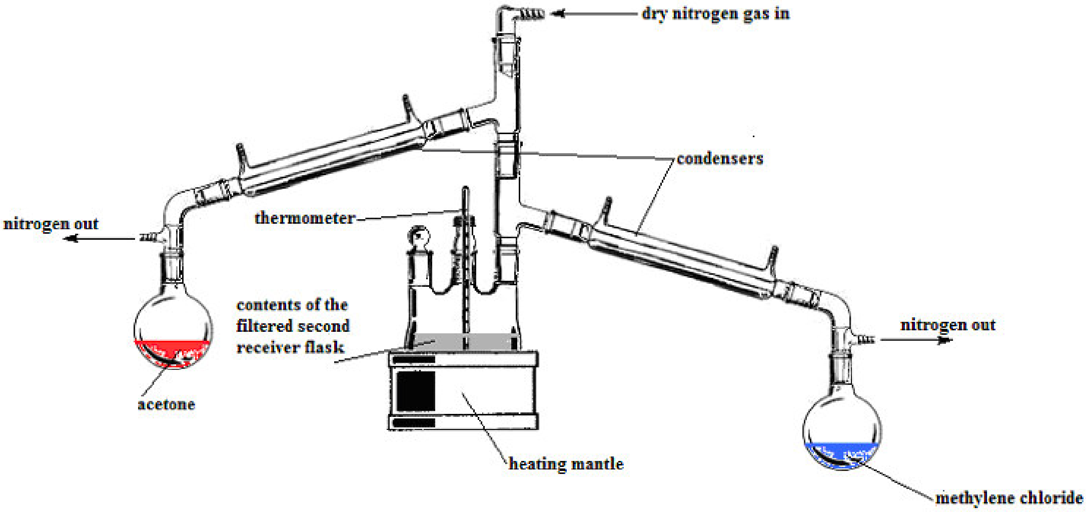
Step 4: Chromatography separation of trisodium hexazinane-1,3,5-triide, and trisodium hexazinane-1,2,4-triide to collect and purify the desired trisodium hexazinane-1,3,5-triide. Note: this process is long and difficult, and the hardest and most expensive part of this whole procedure.

Into the following setup (follow the outline as shown in the following illustration), pour in 500 milliliters of methylene chloride, followed by 500 milliliters of benzene. Note: turn the stopcocks on the columns so there is a slow drip, and pour in methylene chloride first, until all the methylene chloride has passed through both columns and into the receiver flask. Then do the same with the benzene. Thereafter, pour in 250 milliliters of a 30% ferric chloride hexahydrate solution and allow a slow drip until all the ferric chloride solution has passed through both columns and into the receiver flask. After the methylene chloride, followed by the benzene, and then followed by the ferric chloride hexahydrate solution has passed through the first two columns, remove the receiver flask, replace it with a new one and then slowly pour the contents of the replaced receiver flask back through both columns, allowing a slow drip until all the liquid contents have passed through both columns—repeat this process a total of 3 times. Now, after 3 times, remove the receiver flask and then slowly pour it into columns three and four utilizing a slowly drip, until all the liquid mixture has passed through both columns and into the second receiver flask. Thereafter, pour in slowly, 500 milliliters of acetone, and allow the acetone to slowly pass through columns three and four. After the addition of the acetone, remove the second receiver flask, and replace it with a new one, then slowly pour, utilizing a slow drip the contents of the second receiver flask until all the mixture has passed through both columns three and four. Repeat this process a total of 3 times.


30% ferric chloride hexahydrate solution
methylene chloride followed by benzene
acetone
dried residue from step 3
150 grams of phosphorus pentoxide, and 100 grams of 1mm diameter glass beads
columns
100 grams of fine coarse sand, and 100 grams of boric anhydride
150 grams of tetrapotassium EDTA, 100 grams of lauryl sulfate, and 50 grams of 3mm diameter glass beads
receiver flask
100 grams of triethyl amine hydrochloride
small glass sieves
columns three and four
200 grams of aluminum oxide
96 grams of potassium hydroxide beads
second receiver flask

Setup for chromatography separation of trisodium hexazinane-1,3,5-triide, and trisodium hexazinane-1,2,4-triide.

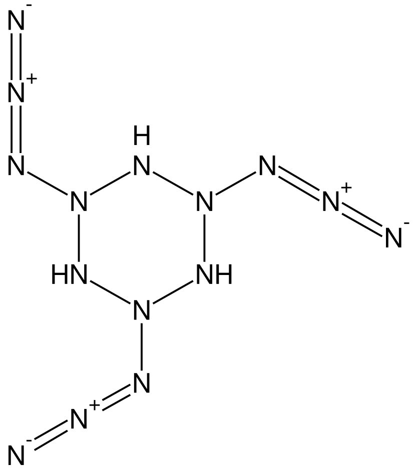
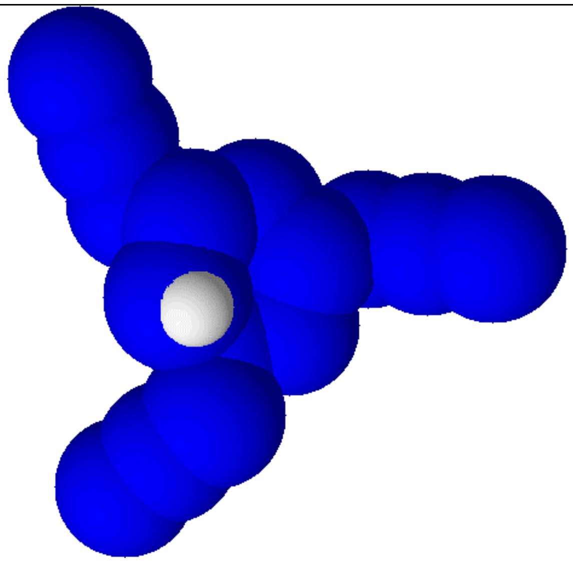
Finally, after 3 times, remove the second receiver flask, and quickly filter it to remove any insoluble impurities. Now, assemble the apparatus as illustrated below, and then place the contents of the filtered second receiver flask into the apparatus, and heat the mixture to 75 Celsius at STP. Continue to distill the mixture until no more methylene chloride or acetone distills over.



2-path distillation apparatus to separate methylene chloride and acetone from the mixture.

When no more acetone or methylene chloride distills over remove the heat source and then immediately disassemble the distillation apparatus, and suction filter the remaining mixture from the 3-neck flask before it cools. After the filtration, pour the filtered mixture into a shallow pan, and allow it to cool to room temperature. Once it has cooled to room temperature, pour over the cooled mass, 750 milliliters of 99% hexane, and then allow the contents in the shallow pan to settle and stand at room temperature for 7 days. After 7 days, scrape up the entire mass covering the shallow pan, and place into a large beaker, the boil the contents in the beaker at 70 Celsius, and when the temperature of the contents of the beaker reaches 70 Celsius, quickly suction filter the hot mixture to remove insoluble impurities. Immediately after the filtration, pour the filtered contents into a rotary evaporator and distill off the benzene and hexane under high vacuum and at a temperature around 70 Celsius until dry solid remains. When dry solid remains, stop the evaporation process, and then remove the dried left over residue. This residue will contain 95% trisodium hexazinane-1,3,5-triide. Recrystallization from hexane several times will purify it to about 98.2%, which is plenty pure for use as an explosive. Dissolving the 98.2% product into mercury followed by filtration, followed by vacuum distillation to remove the mercury, forms 99.8% pure product.

Theoretical Preparation 3: 3,3',3''-hexazinane-1,3,5-triyltris(triaza-1,2-dien-2-ium-1-ide); HEXAAZIDE; HTA

 3,3',3''-hexazinane-1,3,5-triyltris(triaza-1,2-dien-2-ium-1-ide)	
Expanded structure	3D structure

Molecular data:

Molecular Formula = H_3N_{15}
Formula Weight = 213.12432
Composition = H(1.42%) N(98.58%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 213.069585 Da
Nominal Mass = 213 Da
Average Mass = 213.1243 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
Unknown; estimated to be 6500 meters per second	Flammable solid; In oxygen, it burns with a green flame producing fumes of ammonia, nitrogen dioxide, water, and nitrogen. In the absence of air, it burns forming ammonia, and nitrogen gases burning with a yellow flame.	Stable to shock, percussion, and friction; however, samples will detonate when struck with a large hammer under 1000 psi, or when struck by a rifle bullet. Requires blasting cap or detonator for initiation. A secondary explosive.	Stable at room temperature and temperatures up to 180 Celsius;	Colorless to white crystalline mass or powder. Stable at room temperature. Slowly hydrolyzes in moist air, so should be stored in airtight glass container in a cool dark place. Melting point: 121 Celsius, Boiling point: decomposes above 280+ Celsius. In soluble in water (but is slowly hydrolyzed by hot water forming the dangerous hydrazoic acid); barely soluble in methylene chloride, hexane, carbon tetrachloride, and ether. Soluble in liquid ammonia.

Estimated Compound summary: 3,3',3''-hexazinane-1,3,5-triyltris(triaza-1,2-dien-2-ium-1-ide) shows remarkable properties as it has a massive 98.58% nitrogen content. The compound detonates producing pure gaseous ammonia vapor and nitrogen, and has tremendous heaving power making it well suitable for rock blasting, mining, road clearing, rockets and missiles, and a component of mortar, artillery and high velocity tank shells. Because it burns cleaning, it could be used as a high performance rocket fuel either by itself directly, or when combined with powered metals like aluminum, iron, or, magnesium. Note: due to its high heat of deflagration, this substance should not be used in air inflation devices such as emergency rafts, or automobile air bags, as heats of reaction could melt the materials and cause fires. **Warning!** 3,3',3''-

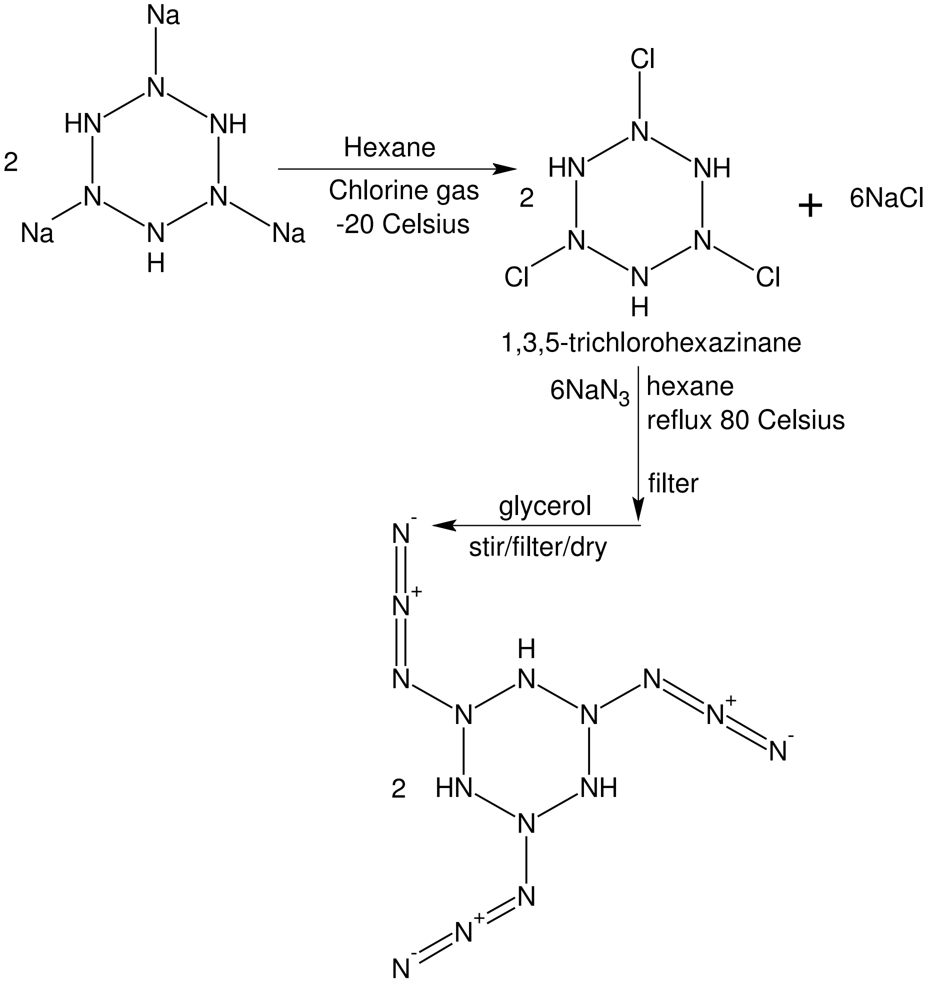
hexazinane-1,3,5-triyltris(triaza-1,2-dien-2-ium-1-ide) is a deadly poison, with an LD_{50} of 9.2 mg per kilogram of body weight. Handle with gloves, and use a respirator to avoid inhaling any dust or small particles.

Safety Info: Hexane is highly flammable so extinguish any source of ignition. Use proper ventilation and avoid inhalation when handling chlorine gas, which is toxic. Use proper ventilation and extinguish all sources of ignition when handling dry ether, as it is highly flammable: Note: use caution when handling ether that has been stored for some prolonged time as it can absorb oxygen forming dangerous peroxides which explode when heated. Before using ether that has been in storage for prolonged periods of time perform a peroxide test in the usual manner. Wear gloves when handling sodium azide and avoid inhalation of dust and/or particles, as sodium azide is highly toxic.

Materials:

1. 126 grams trisodium hexazinane-1,3,5-triide (obtained in theoretical preparation 2, step 4)	5. 157 grams of sodium azide
2. 2170 milliliters of hexane	6. 1500 milliliters of glycerol (pre-heated to 50 Celsius),
3. 171 grams a dry chlorine gas	

Reaction Equation:

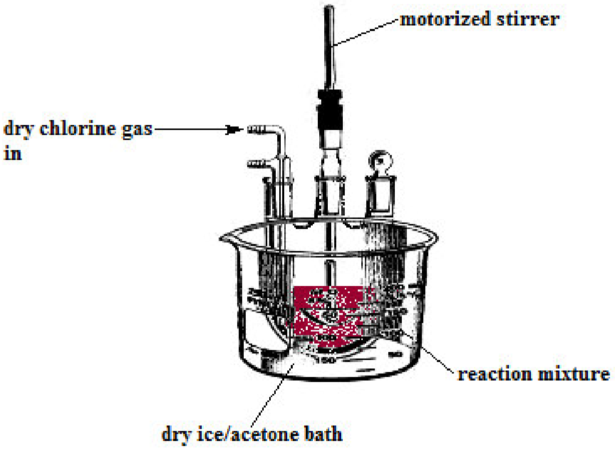


3,3',3''-hexazinane-1,3,5-triyltris(triaza-1,2-dien-2-ium-1-ide)

Preparation:

Step 1: Preparation of 1,3,5-trichlorohexazinane

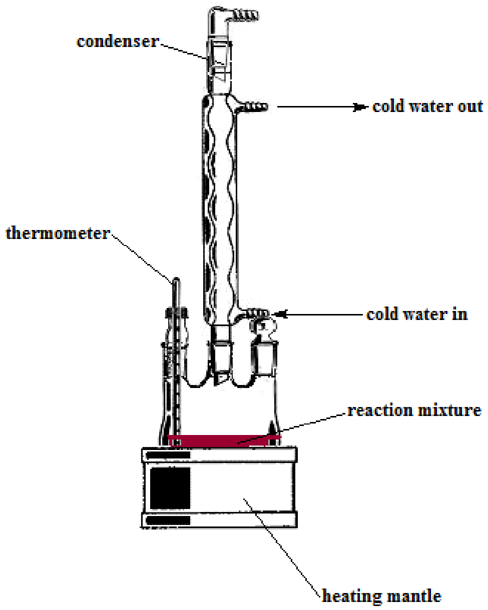
Into a suitable apparatus as illustrated below, place 126 grams trisodium hexazinane-1,3,5-triide (obtained in theoretical preparation 2, step 4), and then pour in 750 milliliters of hexane. Thereafter, begin to stir the mixture, and then bubble into the mixture 171 grams a dry chlorine gas (use a fume hood with good ventilation) at a flow rate of about 500 mg/per minute. Continue to bubble in the chlorine gas while stirring the reaction mixture and maintaining its temperature below –15 Celsius at all times. After the addition of the chlorine, continue to stir the reaction mixture at a temperature below –15 Celsius for about 30 minutes. After 30 minutes, stop stirring the reaction mixture, and then disassemble the apparatus, remove the dry/ice acetone bath, and then suction filter the reaction mixture to remove the insoluble by-products. Thereafter, place the filtered reaction mixture into a distillation apparatus and distill-off the hexane at 70 Celsius until dry residue remains. Once dry residue remains, recrystallize the residue from 750 milliliters of hexane, and then collect the 1,3,5-trichlorohexazinane using suction filtration, and then dry in a an oven at 100 Celsius for 4 hours. After 4 hours, remove the dried 1,3,5-trichlorohexazinane and save for step 2.



Setup for step 1.

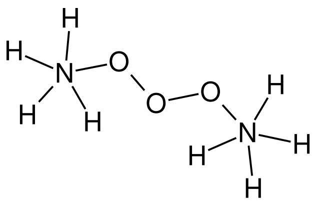
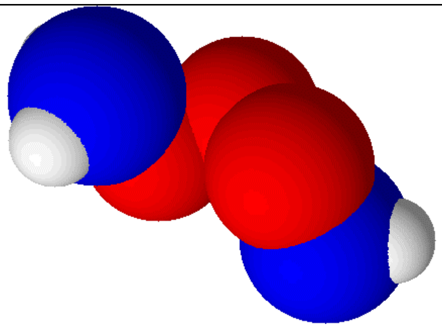
Step 2: Preparation of 3,3',3''-hexazinane-1,3,5-triyltris(triaza-1,2-dien-2-ium-1-ide)

Into a suitable sized 3-neck flask, equipped with powder addition funnel and motorized stirrer, place the dried residue from step 1, and then pour in 670 milliliters of dry hexane. Thereafter, begin to add in small portions at a time (1 gram per portion) of sodium azide to the reaction mixture with vigorous stirring until a total of 157 grams of sodium azide has been added. Note: sodium azide is poisonous so wear gloves and respirator to avoid skin contact or inhalation, After the addition of the sodium azide, assemble the apparatus as illustrated below, and then reflux the reaction mixture at 80 Celsius with vigorous stirring for 6 hours. After 6 hours, remove the heat source, and then allow the refluxed reaction mixture to cool to room temperature, then disassemble the apparatus, and then suction filter the reaction mixture to remove the insoluble product and the by-product sodium chloride, and then suction dry as best as possible. Now, take this suction dried filtered-off mass, and add it to a clean beaker filled with 1500 milliliters of glycerol (pre-heated to 50 Celsius), and then stir this new mixture for 1 hour to dissolve the by-product sodium chloride. After stirring for 1 hour, filter-off the insoluble desired product using suction filtration, wash with three 750-milliliter portions of cold water, and then suction dry as best as possible. Finely, place this washed product into an oven and bake at 105 Celsius for 12 hours. After 12 hours, turn off the oven, and allow the dried product to cool to room temperature. Thereafter recover the desired 3,3',3''-hexazinane-1,3,5-triyltris(triaza-1,2-dien-2-ium-1-ide), and store it in a airtight glass jar in a cool dry place until use. Note: If desired, you can purify the product by dissolving it into liquid ammonia, filtering, and then evaporating off the ammonia. This process will produce a product of 99% purity.



Setup for refluxing the reaction mixture.

Theoretical Preparation 4: diammonium trioxidane-1,3-diide; diammonium trioxide; DATD

 H H N O H H O O H N H H H diammonium trioxidane-1,3-diide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_8N_2O_3$
Formula Weight = 84.0581428
Composition = H(7.37%) N(34.14%) O(58.49%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 82.036745 Da
Nominal Mass = 82 Da
Average Mass = 82.0581 Da

Molecular Information:

Detonation velocity	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

(estimated by monoisotopic mass and index of refraction):				
Estimated: 2350	Non-flammable; burns violently when mixed with reducing agents such as sulfur, charcoal, phosphorus, powdered metals, and hypophosphites.	Very stable at room temperature but dissociates when strongly heated into ammonia, water, and nitrogen. Requires blasting cap or detonator for initiation. A secondary explosive	Strong oxidizer; oxidizes primary alcohols to carboxylic acids; converts secondary alcohols to ketones; Oxidizes sulfites to sulfates; phosphites to phosphates; reacts violently with powered metals when heated or touched by a spark or flame. Explodes in contact with metallic sodium or metallic potassium, when warmed or heated. Oxidizes ethers to peroxides; Oxidizing activity and capabilities very similar to potassium permanganate i.e., reacts with toluene converting the methyl group to carboxylic acid.	Colorless to white crystalline cubic crystals or fine free flowing powder. Melting point: 189 Celsius; decomposes when heated above 210 Celsius. 1 gram dissolves in 2.3 milliliters of water. Practically insoluble in benzene, hexane, and most organic solvents.

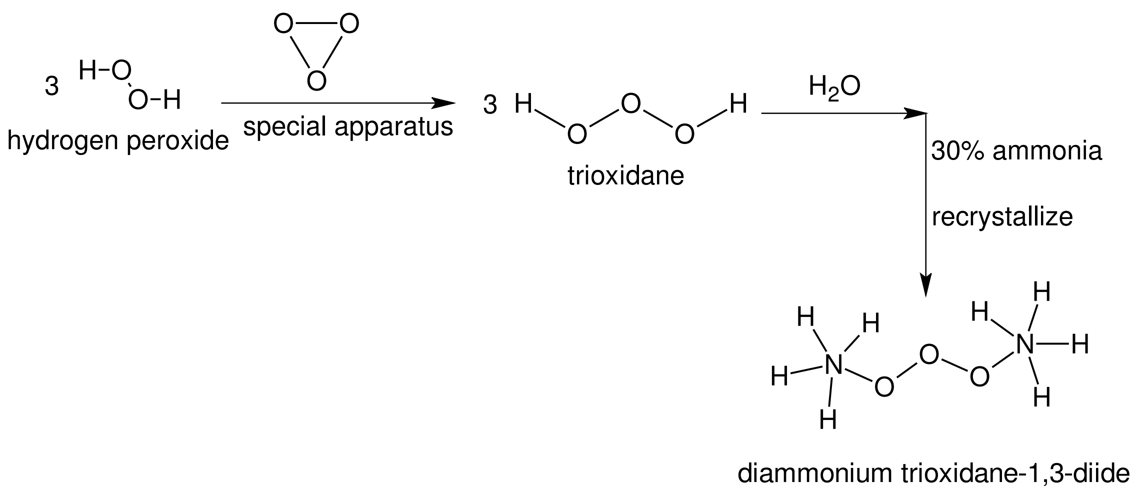
Estimated Compound summary: diammonium trioxidane-1,3-diide is a strong oxidizer similar to ammonium nitrate and potassium permanganate. Its low detonation energy makes it useful in blasting compositions for blasting soft rock, clay, soil, and dirt. diammonium trioxidane-1,3-diide can replace ammonium nitrate in ANFO, forming DATFO compositions containing 94% diammonium trioxidane-1,3-diide and 6% fuel oil—this mixture can be used to fill cardboard or plastic tubes for safe mining operations mainly for commercial purposes, and diammonium trioxidane-1,3-diide can replace other ammonium nitrate blasting agents. diammonium trioxidane-1,3-diide has potential use as an oxidizing agent in solid rocket fuels when mixed with reducing agents and cured with a plasticizer and epoxy, and these rocket fuels have greater energy then ammonium nitrate based rocket fuels. diammonium trioxidane-1,3-diide can also be used to make pyrotechnic mixtures for making flares, fireworks, tracers, and many other pyrotechnic applications. Its use in making fireworks is a remarkable new era of new firework pyrotechnic formulations that need vast testing, which is currently not undergoing.

Safety Info: Use proper care when handling 90% hydrogen peroxide, which can often be unstable especially in contact with metals, sunlight, dust, and many organic substances. Use proper ventilation and wear a respirator when handling ozone gas, which is toxic and corrosive. Use proper ventilation when handling concentrated ammonia solution.

Materials:

1. 190 grams of 90% hydrogen peroxide	3. 1100 milliliters of distilled water
2. 80 grams of dry ozone gas (from tank)	4. 550 grams of a 30% concentrated aqueous ammonia solution

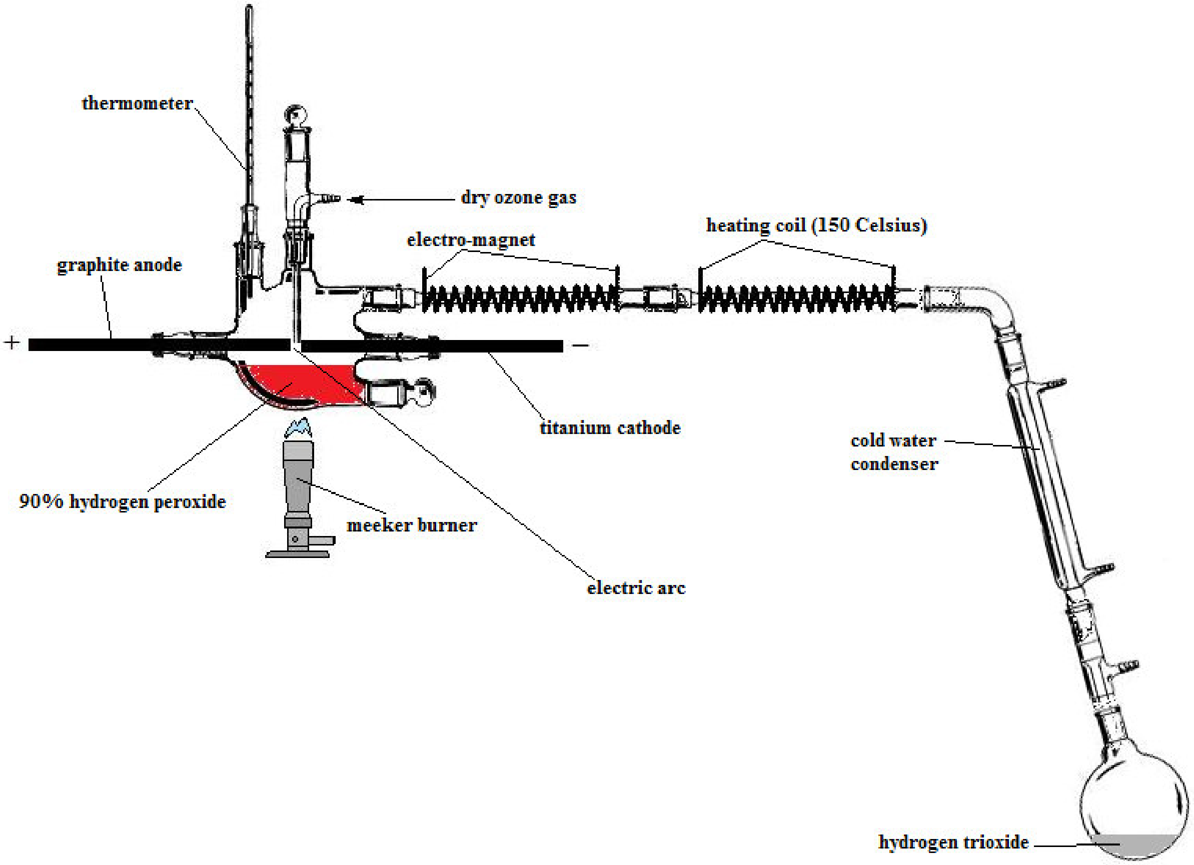
Reaction Equation:



Preparation:

Step 1: Preparation of hydrogen trioxide (trioxidane)

Assemble the special apparatus as illustrated below. Note: This apparatus will have to be specially ordered and manufactured. Thereafter, pour in 190 grams of 90% hydrogen peroxide. Thereafter, completely flush out the entire apparatus with argon gas before proceeding. Then, using a power supply, attach the black negative clamp to the titanium cathode, and the red positive clamp to the graphite anode, and then turn on the power supply to 50,000 volts, at 0.1 amp. Thereafter, begin to heat the 90% hydrogen peroxide to 160 Celsius using a meeker burner. Note: removing the burner on and off will be needed in order to maintain a proper temperature. When the temperature inside the apparatus reaches around 160 Celsius, begin to pump in dry ozone gas at a flow rate of 500 mg per/minute. Thereafter, continue to pump in dry ozone gas until 80 grams of dry ozone gas has been added. After the addition of the ozone gas is complete, turn off the power source to the electrodes, and continue to heat the apparatus using the meeker burner at 160 Celsius, removing it on and off to maintain the temperature inside the reaction apparatus at around 160 Celsius until all liquid in the reaction apparatus has distilled over. When this point is reached remove the heat source, and then allow the reaction apparatus to cool to room temperature. Note: Also, remove the heat source to the heating coil wrapped around the second tube as illustrated below. Thereafter, remove the receiver flask containing the hydrogen trioxide and save for step 2; this product will contain about 5% water. Note: Hydrogen trioxide is a deep blue, syrupy liquid, with a melting point of –34 Celsius, and a boiling point of 197 Celsius. Decomposes when heated above 380 Celsius.

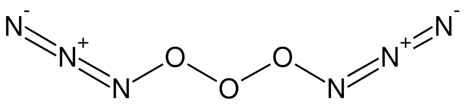
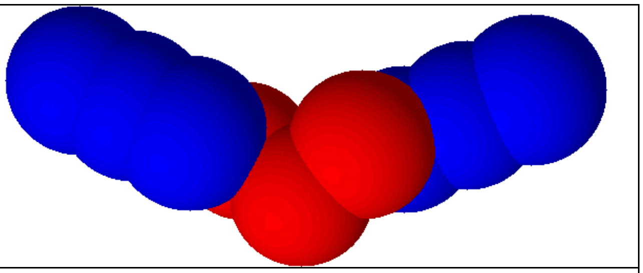


Setup apparatus for preparation of hydrogen trioxide.

Step 2: Preparation of diammonium trioxidane-1,3-diide

Into a suitable sized beaker pour in the hydrogen trioxide prepared in step 1, and then slowly pour in 500 milliliters of distilled water. Thereafter stir the liquid mixture to form a uniform mixture. Thereafter, slowly pour into the beaker 550 grams of a 30% concentrated aqueous ammonia solution while stirring the reaction mixture. After the addition of the concentrated ammonia, stir the reaction mixture at room temperature for about 1 hour. Afterwards, recrystallize the desired diammonium trioxidane-1,3-diide from the reaction mixture, and collect the precipitated crystals using suction filtration. Thereafter, dissolve the collected crystals into 600 milliliters of distilled water, and then recrystallize the desired product from this aqueous mixture, and collect the crystals using suction filtration. Thereafter, dry the crystals in an oven at 100 Celsius for 6 hours.

Theoretical Preparation 5: 3,3'-trioxidane-1,3-diylbis(triaza-1,2-dien-2-ium-1-ide); TDTD

 3,3'-trioxidane-1,3-diylbis(triaza-1,2-dien-2-ium-1-ide)	
Expanded structure	3D structure

Molecular data:
Molecular Formula = N_6O_3
Formula Weight = 132.0384
Composition = N(63.65%) O(36.35%)

Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 132.003188 Da
Nominal Mass = 132 Da
Average Mass = 132.0384 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
Estimated: 3700	Non-Flammable.	Stable. Can be stored for years without any danger. Decomposes non-violently when heated to 300 Celsius. Impact sensitivity = 1 kilogram drop head from 6 meters. Requires blasting cap or detonator for initiation. A secondary explosive.	Non-reactive.	Light lemon-yellow needle like crystals from methanol. Light orange yellow crystals from water. Melting point: 89 Celsius; boiling point: decomposes. Moderately soluble in water, alcohol, and ether, but practically insoluble in most organic solvents. **Warning!** Very toxic. Do not handle with bare hands. LD_{50} 12 mg per kilogram.

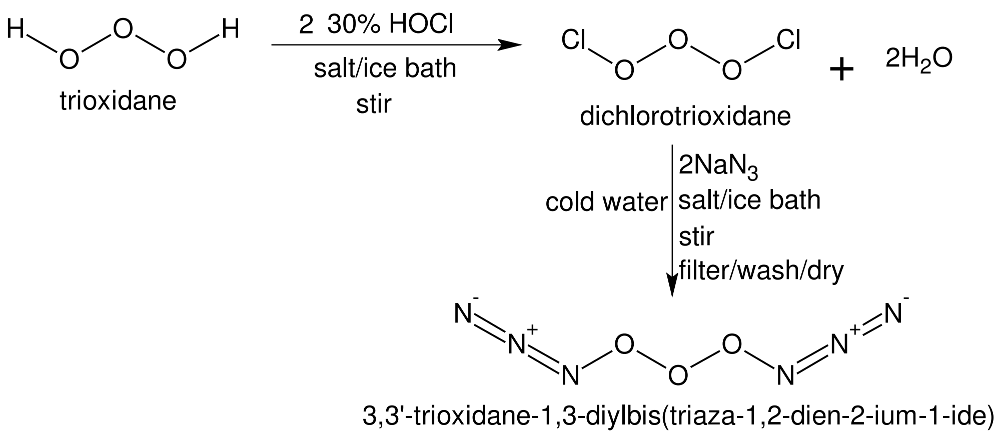
Estimated Compound summary: 3,3'-trioxidane-1,3-diylbis(triaza-1,2-dien-2-ium-1-ide) is a moderately powerful explosive with a high degree of use in rocket fuels, high performance gun propellants, and blasting charges for mining soft rock, sedimentary deposits, and for demolition work. 3,3'-trioxidane-1,3-diylbis(triaza-1,2-dien-2-ium-1-ide) can easily be melted and casted into shape charges when admixed with TNT to form moderately penetrating munitions for use in shoulder fired rockets for anti-bunker, anti-machine gun nests, guard towers, and anti-pill box operations.

Safety Info: Wear gloves when handling sodium azide, as it is highly toxic. Wear gloves and avoid ingestion or inhalation of methanol, which is poisonous.

Materials:

1. 250 grams of hydrogen trioxide (prepared in theoretical preparation 4, step 1)	3. 812 grams of sodium azide
2. 2500 milliliters of 30% hypochlorous acid solution	

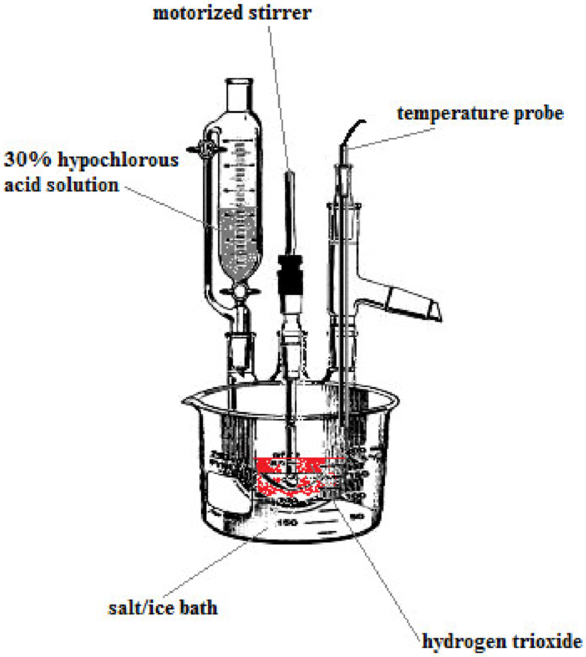
Reaction Equation:



Preparation:

Step 1: Preparation of dichlorotrioxidane

Into a suitable apparatus as illustrated, place 250 grams of hydrogen trioxide (same product theoretical preparation 4, step 1), and then place the reaction flask into a salt/ice bath. When the temperature of the hydrogen trioxide reaches –5 to 0 Celsius, slowly add in, drop by drop of a 30% hypochlorous acid solution while vigorously stirring the hydrogen trioxide reaction mixture and maintaining its temperature around –5 to 0 Celsius. Keep adding the hypochlorous acid solution to the hydrogen trioxide reaction mixture until 2500 milliliters of 30% hypochlorous acid solution has been added. After the addition of the hypochlorous acid solution, continue to stir the reaction mixture at a temperature around 0 Celsius for 2 hours. After 2 hours, disassemble the apparatus, and pour the reaction mixture into a suitable sized beaker, and then immediately go to step 2.

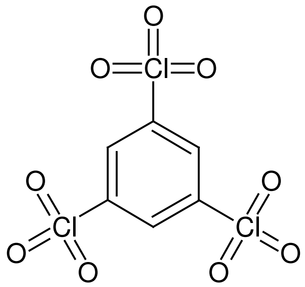
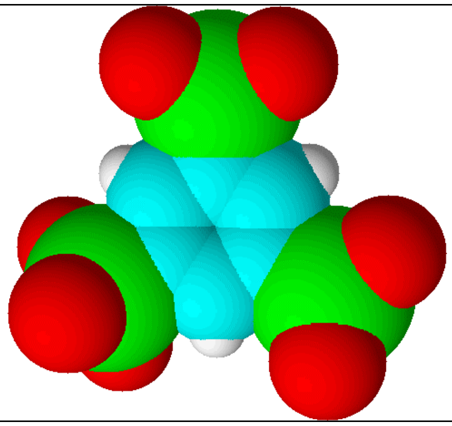


Setup for preparation of dichlorotrioxidane.

Step 2: Preparation of 3,3’-trioxidane-1,3-diylbis(triaza-1,2-dien-2-ium-1-ide)

To the dichlorotrioxidane contained in the beaker from step 1, add in 1000 milliliters of pre-chilled ice water, and then place the beaker into an ice/salt bath and allow it to chill to –5 to 0 Celsius. Note: putting the beaker in a freezer can speed up the cooling process. When the temperature of the dichlorotrioxidane reaches –5 to 0 Celsius, add in, in small portions at a time, 812 grams of sodium azide, while vigorously stirring the dichlorotrioxidane reaction mixture, and maintaining its temperature at –5 to 0 Celsius at all times. After the addition of the sodium azide, continue to stir the reaction mixture at temperature of –5 to 0 Celsius for 6 hours. After 6 hours, remove the salt/ice bath, and allow the reaction mixture to warm to room temperature. Thereafter, filter-off the insoluble desired product using suction filtration, wash with three 750-milliliter portions of hot water, and then suction dry as best as possible. Finely, place this suction dried product into a dessicator over anhydrous calcium chloride for 14 days before using.

Theoretical Preparation 6: benzene-1,3,5-triyltris(chlorane) nonaoxide; BTCN; Chlorane

 benzene-1,3,5-triyltris(chlorane) nonaoxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6H_3Cl_3O_9$
Formula Weight = 325.44162
Composition = C(22.14%) H(0.93%) Cl(32.68%) O(44.25%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 323.884265 Da
Nominal Mass = 324 Da
Average Mass = 325.4416 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7570	Flammable; burns with a black smoky flame when pure emitting hydrogen chloride, carbon monoxide, carbon dioxide, water and carbon, but often flashes when admixed with powdered metals	Non-sensitive. Can be stored for years without any danger. A small sample can be detonated when struck with a rifle bullet. Requires blasting cap or detonator for initiation. A secondary	Stable. May explode in contact with alcohols if heated.	Colorless to white crystalline masses or powder. Melting point: 167 Celsius when pure. When impure may vary from 121 to 134 Celsius. Boiling point: decomposes

	and ignited.	explosive.		above 398 Celsius. Insoluble in water, and most organic solvents, but soluble in liquid ammonia and concentrated sulfuric acid.

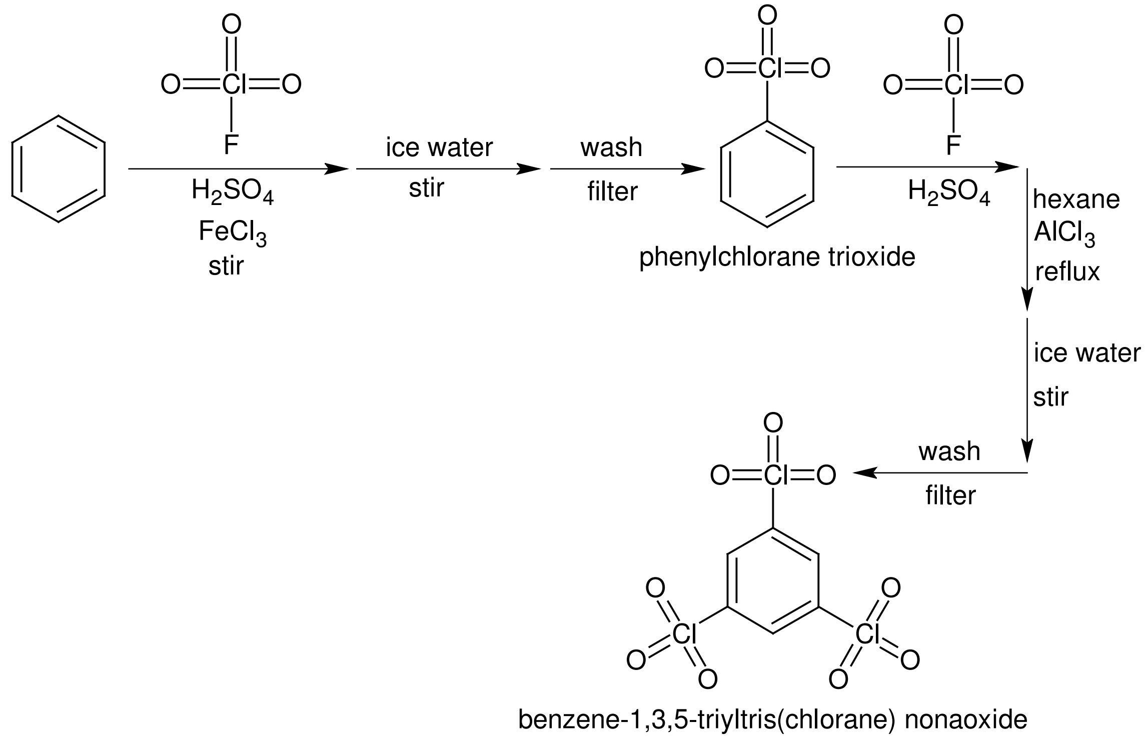
Estimated Compound summary: benzene-1,3,5-triyltris(chlorane) nonaoxide is a radical new explosive in the nitro sense of things, whereby chlorine occupies the nitrogen slot. Its highly oxidized chlorine atoms have remarkable energy, and thus this substance is more powerful then its nitrogen counter part: 1,3,5-trinitrobenzene. benzene-1,3,5-triyltris(chlorane) nonaoxide can be used in place of nitrobenzenes, for use in all sorts of explosives munitions including bombs, artillery shells, mortar rounds, high explosive projectiles of all kinds and benzene-1,3,5-triyltris(chlorane) nonaoxide shows remarkable use in making high performance gun propellants primarily when mixed with nitrocellulose, a lacquer and then extruded into any desired grain size or shape and then allowed to cure. Due to its stability, it is very useful for manufacturing warheads for high velocity munitions such as tank-fired projectiles. It can also be melted and casted into powerful shape charges for use in anti-tank weapons, cutting for demolitions operations, or any desired penetrating means.

Safety Info: Use proper ventilation when handling benzene and avoid inhalation and skin contact, as benzene is a carcinogen. Wear gloves and use caution when handling concentrated sulfuric acid, which is highly corrosive and can cause skin burns. Use a fume hood with good ventilation when handling perchloryl fluoride gas. Avoid inhalation and skin contact of perchloryl fluoride, which is toxic. Use extreme care when handling perchloryl fluoride, which explodes in contact with many organic substances such as alcohols. Wear gloves when handling anhydrous aluminum chloride, which reacts violently with water forming hydrogen chloride fumes.

Materials:

1. 191 grams of dry benzene	5. 3500 milliliters of a 5% sodium carbonate solution
2. 850 milliliters of concentrated sulfuric acid	6. 750 milliliters of dry hexane
3. 15 grams of anhydrous ferric chloride	7. 20 grams of anhydrous aluminum chloride
4. 550 grams of perchloryl fluoride gas from tank	8. Dry nitrogen gas from tank

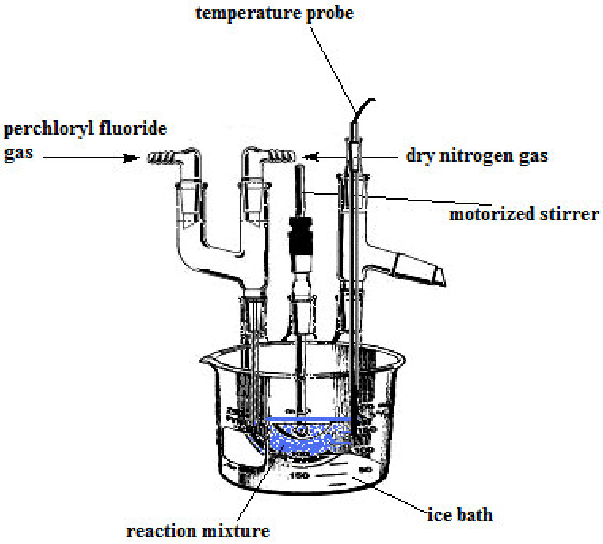
Reaction Equation:



Preparation:

Step 1: Preparation of phenylchlorane trioxide

Into a 3-neck flask, equipped with motorized stirrer, thermometer, and gas inlet tube, pour in 191 grams of dry benzene. Thereafter, slowly add in 350 milliliters of concentrated sulfuric acid. Thereafter, add in, in small portions, 15 grams of anhydrous ferric chloride, and then place the 3-neck flask into an ice bath as illustrated below, and then begin to stir the contents of the 3-neck flask. Now, when the temperature of the reaction mixture reaches 5 to 0 Celsius, begin a slow addition of perchloryl fluoride gas admixed with dry nitrogen gas at a flow rate of about 100 mg per minute, while rapidly stirring the reaction mixture and maintaining its temperature below 10 Celsius. Continue to bubble in the perchloryl fluoride gas until a total of 249 grams of perchloryl fluoride gas has been added. Note: flush the entire apparatus with dry nitrogen gas prior to the addition of perchloryl fluoride. After the addition of the perchloryl fluoride gas, continue to stir the reaction mixture at a temperature of 5 to 0 Celsius for 30 minutes. After 30, minutes disassemble the apparatus, slowly and carefully pour the reaction mixture into a large beaker containing ice water, and then add more ice, and then stir the entire mixture for about 1 hour. Note: use caution when pouring the reaction mixture into the ice water as sulfuric acid generates tremendous heat when diluted with water. Make sure to use a large enough beaker to allow for room for more ice to be added to cool the reaction mixture. After stirring the mixture for 1 hour, filter off the insoluble solid, which will be the desired phenylchlorane trioxide, and then wash the phenylchlorane trioxide with 1500 milliliters of a 5% sodium carbonate solution followed by washing with 1500 milliliters of hot water using suction filtration, and then suction dry the washed phenylchlorane trioxide product, and save for step 2.

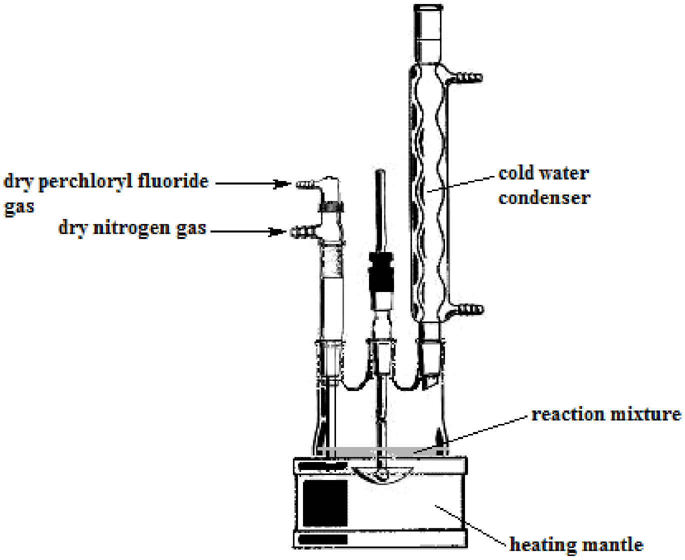


Setup for addition of perchloryl fluoride gas.

Step 2: Preparation of benzene-1,3,5-triyltris(chlorane) nonaoxide

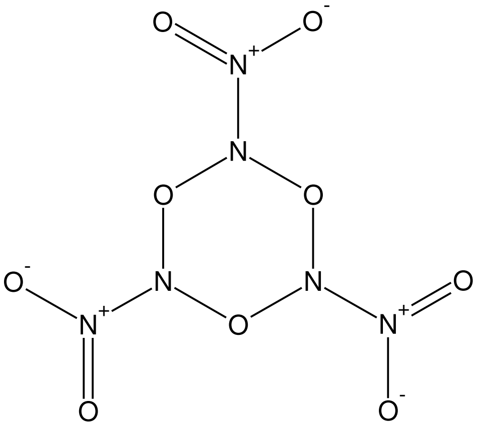
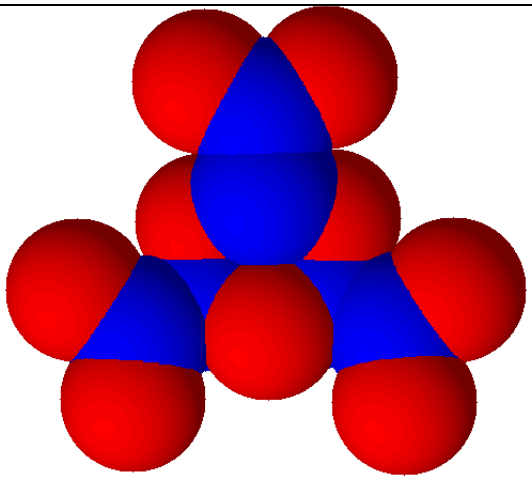
Into an apparatus as illustrated below (using a 4-neck flask for thermometer adapter, not pictured), pour in 750 milliliters of dry hexane, followed by 500 milliliters of concentrated sulfuric acid, and then add in the phenylchlorane trioxide product obtained from step 1 and stir the entire mixture until the phenylchlorane trioxide dissolves in the concentrated sulfuric acid. Thereafter, add in 20 grams of anhydrous aluminum chloride, and then turn on the heating mantle to a temperature of 70 Celsius. When the contents in the 4-neck flask reach 70 Celsius, begin to bubble into the reaction mixture perchloryl fluoride/dry nitrogen gas at a flow rate of 100 mg per/minute while rapidly stirring the reaction mixture and maintaining its temperature around 70 Celsius. Continue to bubble into the reaction mixture perchloryl fluoride/nitrogen gas until a total of 301 grams of perchloryl fluoride has been added. After the addition of the perchloryl fluoride gas, continue to rapidly stir the reaction mixture and reflux it at a temperature around 70 Celsius for 1 hour. After 1 hour, stop the refluxing process, and then remove the heat source and allow the reaction mixture to cool to room temperature. When the reaction mixtures temperature has been reduced to around room temperature, slowly, and carefully pour the reaction mixture into a large beaker filled with ice water. Note: Sulfuric acid generates tremendous heat when added to water, so use caution, and use a large enough beaker to allow for addition of further ice to keep the mixture at a cool temperature. After the addition of the reaction mixture to the ice water, add more ice, and then stir the entire mixture for 1 hour. After 1 hour, filter-off the insoluble solid, which will be the desired benzene-1,3,5-

triyltris(chlorane) nonaoxide, using suction filtration, and then wash this benzene-1,3,5-triyltris(chlorane) nonaoxide with 2000 milliliters of a 5% sodium carbonate solution, followed by washing with 2000 milliliters of hot water. Thereafter, suction dry the washed benzene-1,3,5-triyltris(chlorane) nonaoxide, and then put the benzene-1,3,5-triyltris(chlorane) nonaoxide into an oven and heat at 105 Celsius for 6 hours. After 6 hours, turn off the oven, and then allow the dried desired product to cool to room temperature. Then, remove the desired product from the oven and store in an amber glass bottle in a cool dry place until use.



Setup for the addition of perchloryl fluoride.

Theoretical Preparation 7: 2,4,6-trinitro-1,3,5,2,4,6-trioxatriazinane; TNTOTA; oxatriazinane

 2,4,6-trinitro-1,3,5,2,4,6-trioxatriazinane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = N_6O_9

Formula Weight = 228.0348

Composition = N(36.85%) O(63.15%)

Molar Refractivity = 34.98 ± 0.4 cm^3

Molar Volume = 86.6 ± 5.0 cm^3

Parachor = 316.8 ± 6.0 cm^3

Index of Refraction = 1.741 ± 0.03

Surface Tension = 178.9 ± 5.0 dyne/cm

Density = 2.63 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 13.86 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 227.972676 Da
Nominal Mass = 228 Da
Average Mass = 228.0348 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
8100	Flammable; burns non-violently with a colorless flame producing water and nitrogen. Ignites violently when mixed with powdered aluminum and ignited.	Non-sensitive. Very stable, can be stored for years. Requires blasting cap or detonator for initiation. A secondary explosive.	Non-reactive.	Slightly yellow-lime crystalline mass or powder. Melting point: 77 Celsius, boiling point: decomposes above 278 Celsius. The compound is insoluble in water, and most organic solvents, but has moderate solubility in methanol.

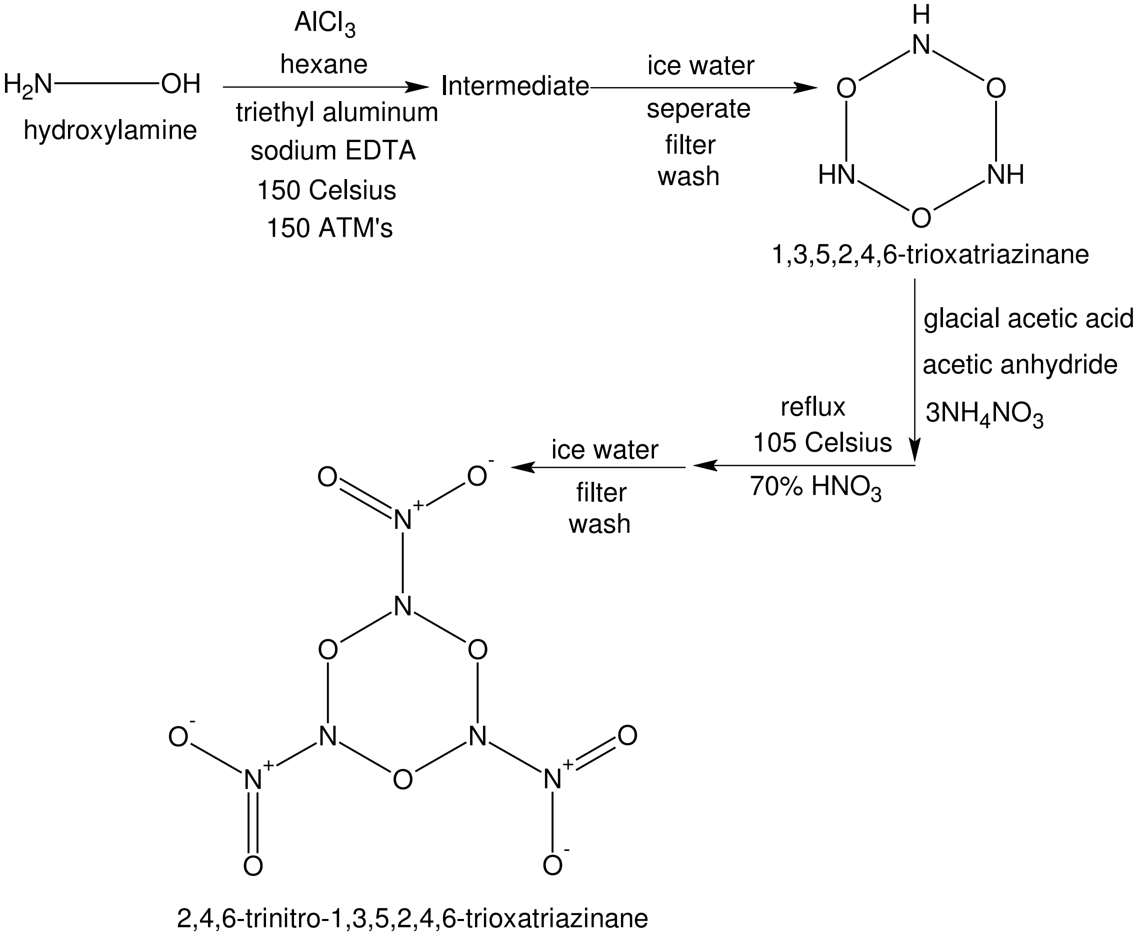
Estimated Compound summary: 2,4,6-trinitro-1,3,5,2,4,6-trioxatriazinane is a high explosive very similar to RDX, and hence can be used in all sorts of explosives operations; however, though its highly unlikely, 2,4,6-trinitro-1,3,5,2,4,6-trioxatriazinane can replace RDX in explosive compositions. Because of its high gas ratio upon ignition, it makes for a perfect high performance rocket fuel especially when admixed with powdered aluminum. Because of its low melting point it can be melted and casted into shape charges with powerful penetrating power.

Safety Info: Extinguish all flames before using hexane, which is highly flammable. Wear gloves when handling hydroxylamine, which is unstable and tends to decompose, keep sealed under nitrogen as it decomposes in moist air. Use extreme caution when handling triethyl aluminum, which is spontaneously flammable in air. Wear gloves when handling anhydrous aluminum chloride and avoid contact with water. Wear gloves when handling glacial acetic acid and acetic anhydride, which are both corrosive. Wear gloves when handling nitric acid, as it is a strong oxidizer and avoid contact with combustible organic material.

Materials:

1. 500 milliliters of dry hexane	6. 2000 milliliters of a 5% sodium carbonate solution
2. 230 grams of hydroxylamine	7. 350 milliliters of glacial acetic acid
3. 50 grams of triethyl aluminum	8. 300 milliliters of acetic anhydride
4. 20 grams of anhydrous aluminum chloride	9. 593 grams of ammonium nitrate
5. 100 grams of sodium EDTA	10. 190 milliliters of 70% nitric acid

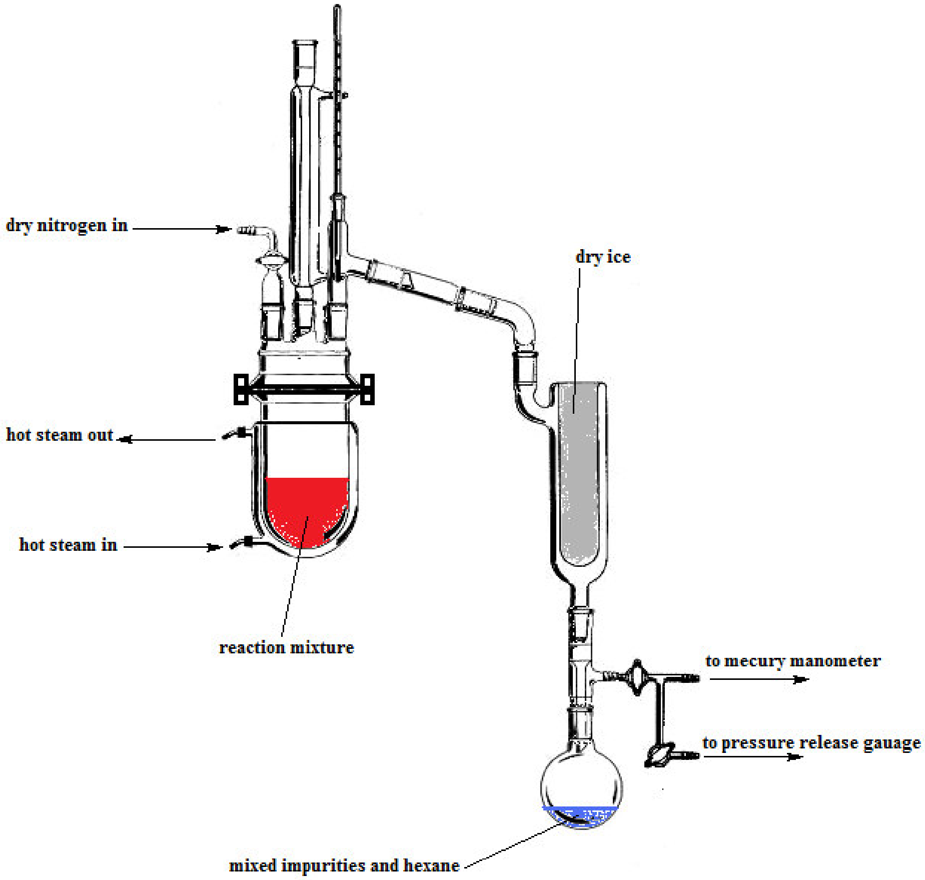
Reaction Equation:



Preparation:

Step 1: Preparation of Intermediate from catalyzed reaction utilizing triethyl aluminum

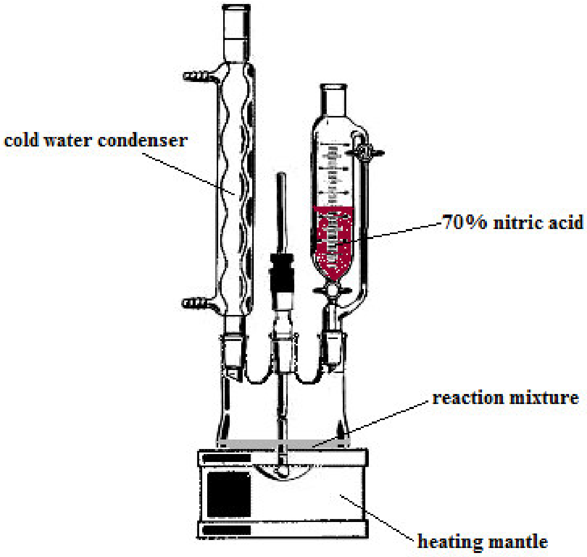
Into the apparatus as illustrated below, place 500 milliliters of dry hexane, followed by 230 grams of hydroxylamine, followed by 50 grams of triethyl aluminum, followed by 20 grams of anhydrous aluminum chloride, and then followed by 100 grams of sodium EDTA. Thereafter, begin a nitrogen purge and continue to purge the apparatus with nitrogen until the pressure gauge reads 150 ATM's. Thereafter, pump super heated steam into the outer casing of the reaction vessel, to heat the contents in the reaction vessel to 150 Celsius. **Warning!** Triethyl aluminum is very dangerous as it spontaneously ignites in air burning with a hot flame, be sure to purge the entire apparatus with dry nitrogen before starting the reaction. Thereafter continue to steam heat and allow the reactants to react under moderate pressure for 6 hours. Now, after six hours, stop the super heated steam system, and allow the contents in the reaction vessel to cool to room temperature. When the reaction mixture has cooled to room temperature remove the nitrogen purge, disassemble the apparatus, and then slowly and gently and carefully pour the reaction mixture into 1500 milliliters of ice water. At this point the triethyl aluminum will decompose, the sodium EDTA will dissolve in the water, and the aluminum chloride will decompose. The hexane will form an insoluble upper layer, which is removed by decantation or by use of a separatory funnel. Left in the ice water after separation of the hexane will be the insoluble intermediate 1,3,5,2,4,6-trioxatriazinane. Now, simply, filter-off the insoluble 1,3,5,2,4,6-trioxatriazinane, wash with 500 milliliters of a 5% sodium carbonate solution, followed by washing with 1000 milliliters of cold water, using suction filtration, and then suction dry this intermediate product of 1,3,5,2,4,6-trioxatriazinane, and save for step 2.



Setup for preparation of intermediate.

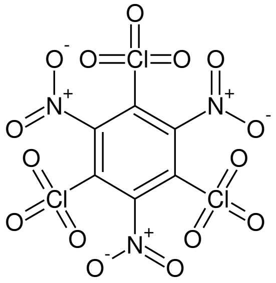
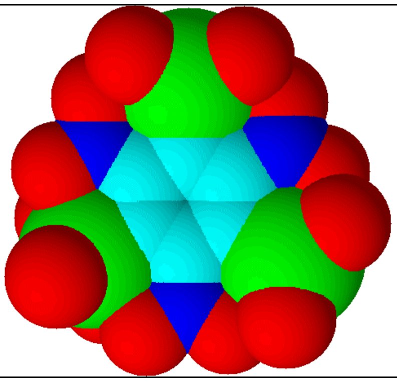
Step 2: Preparation of 2,4,6-trinitro-1,3,5,2,4,6-trioxatriazinane

Into a 3-neck flask as illustrated below, pour in 350 milliliters of glacial acetic acid, followed by 300 milliliters of acetic anhydride. Thereafter, add in 593 grams of ammonium nitrate, followed by the 1,3,5,2,4,6-trioxatriazinane product obtained in step 1. Thereafter, place 190 milliliters of 70% nitric acid into the addition funnel and then heat the reaction mixture to 105 Celsius using the heating mantle, and when its temperature reaches 105 Celsius, begin a slow drip of the 70% nitric acid into the reaction mixture with constant stirring. After all the nitric acid has been added, continue to stir the reaction mixture at a temperature of 105 Celsius for 3 hours. After 3 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Once it has, disassemble the apparatus, and then pour the entire reaction mixture into a large beaker filled with ice. After the ice has melted, add in more ice, and then stir the entire mixture for 1 hour. After 1 hour, filter-off the precipitated product using suction filtration, and then wash with 1500 milliliters of 5% sodium carbonate solution, followed by washing with 1500 milliliters of hot water, then suction dry the desired 2,4,6-trinitro-1,3,5,2,4,6-trioxatriazinane.



Setup for addition of nitric acid to the reaction mixture.

Theoretical Preparation 8: (2,4,6-trinitrobenzene-1,3,5-triyl)tris(chlorane) nonaoxide; Chlorane

 (2,4,6-trinitrobenzene-1,3,5-triyl)tris(chlorane) nonaoxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6Cl_3N_3O_{15}$
Formula Weight = 460.4343
Composition = C(15.65%) Cl(23.10%) N(9.13%) O(52.12%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 458.839499 Da
Nominal Mass = 459 Da
Average Mass = 460.4343 Da

Molecular Information:

Detonation velocity	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

(estimated by monoisotopic mass and index of refraction):				
9790	Flammable solid; burns with a greenish flame emitting toxic fumes of nitrogen oxides, chlorine, and carbon monoxide. Do not detonate inside enclosed areas as toxic fumes will build up and cause potential harm to allied forces. Note: keep this substance away from powered aluminum as explosion will result upon heat, ignition, spark, or flame.	Non-sensitive. Stable at room temperature and temperatures up to 490 Celsius. Requires blasting cap or detonator for initiation. A secondary explosive.	Relatively non-reactive. Can be reduced by heating with a mixture of finely divided tin and concentrated hydrochloric acid.	Orange-yellow to lightly yellowish crystalline mass or powder. Color depends on purity. Impure substance usually has a yellow tint; where as pure substance is mostly an off-white. Melting point: 197 Celsius, Boiling point: Decomposes above 489 Celsius. Insoluble in water, and most organic solvents, but soluble in concentrated sulfuric acid.

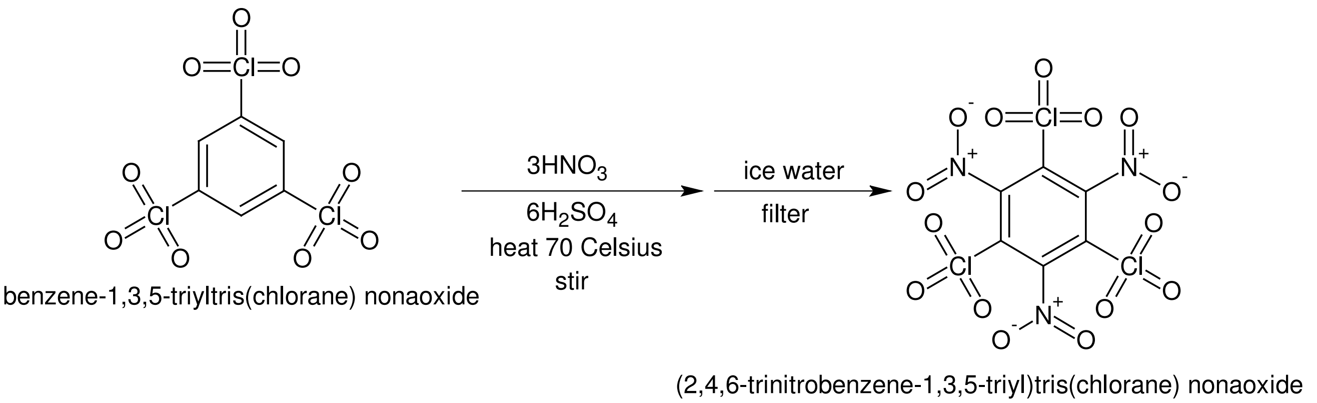
Estimated Compound summary: (2,4,6-trinitrobenzene-1,3,5-triyl)tris(chlorane) nonaoxide is a radical new and fascinating explosive with outstanding physical properties, with tremendous energy. Because it has nitrogen and chlorine atoms in their highest oxidation states this substance has tremendous energy that makes it a powerful substitute to the more common explosives, RDX, TNT, PETN, ect.,; (2,4,6-trinitrobenzene-1,3,5-triyl)tris(chlorane) nonaoxide shows remarkable stability with a relatively high melting point making well suitable for making explosive in space based weapons, high velocity projectiles, rocket warheads, missile warheads, and also because of its stability, it can be used to prepare powerful land mines. (2,4,6-trinitrobenzene-1,3,5-triyl)tris(chlorane) nonaoxide can also be used to prepare high performance gun propellants when admixed with powdered metals with the exception of aluminum.

Safety Info: Use caution and wear gloves and eye protection when handling concentrated nitric and sulfuric acids, both of which are very corrosive. Keep concentrated nitric acid away from copper as poisonous fumes are evolved. Concentrated sulfuric acid is a powerful dehydrating agent that will char many substances including wood.

Materials:

1. 105 grams of 70% nitric acid	3. 126 grams of benzene-1,3,5-triyltris(chlorane) nonaoxide (prepared in theoretical preparation 6, step 2)
2. 200 milliliters of concentrated sulfuric acid	4. 2000 milliliters of a 5% sodium carbonate solution

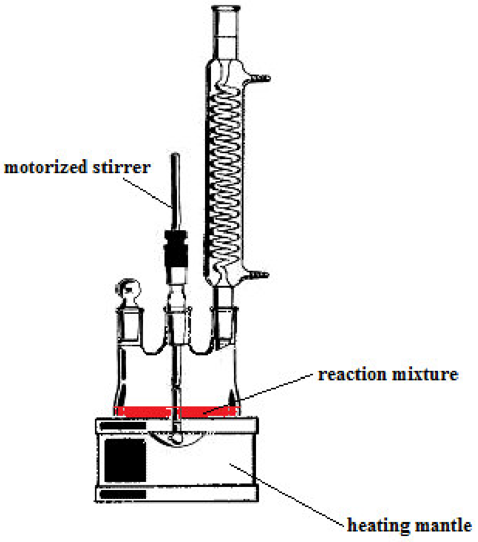
Reaction Equation:



benzene-1,3,5-triyltris(chlorane) nonaoxide

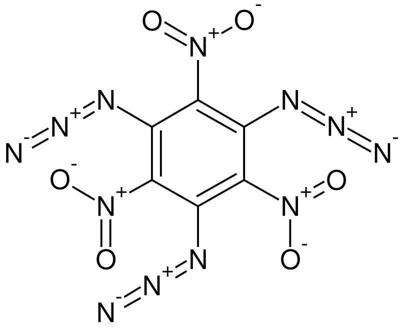
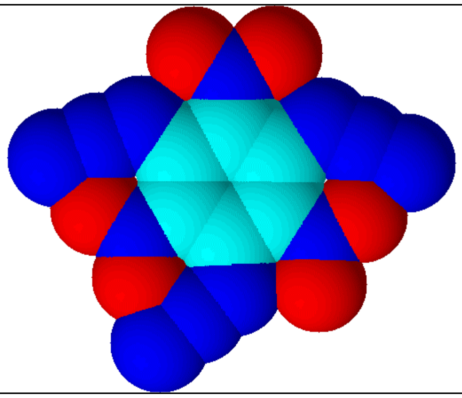
(2,4,6-trinitrobenzene-1,3,5-triyl)tris(chlorane) nonaoxide

Preparation: Into a suitable apparatus as illustrated below, add in 105 grams of 70% nitric acid, and then slowly and carefully pour in 200 milliliters of concentrated sulfuric acid. Note: use caution when adding the sulfuric to the nitric. Afterwards, add in, in small portions at a time, 126 grams of benzene-1,3,5-triyltris(chlorane) nonaoxide (prepared in theoretical preparation 6, step 2). Once all the benzene-1,3,5-triyltris(chlorane) nonaoxide has been added, heat the mixture to 70 Celsius using a heating mantle, and then rapidly stir the reaction mixture at 70 Celsius for about 4 hours. After 4 hours, remove the heat source, and continue to rapidly stir the turbid mixture rapidly until it cools to room temperature. Once it has cooled to room temperature, disassemble the apparatus, and then slowly and cautiously pour the whole reaction mixture into a large beaker filled to the top with ice. Once the ice has melted, rapidly blend the entire mixture, and then add in more ice, and allow it to stand at room temperature until the ice melts. Thereafter, simply filter-off the precipitated product, using suction filtration, and then wash this collected precipitate with 2000 milliliters of a 5% sodium carbonate solution, followed by three 500 milliliter portions of warm water, and then suction dry as best as possible. Thereafter, place the washed suction dried product into an oven and bake at 100 Celsius for 6 hours. After 6 hours, turn off the oven, and allow the dried desired product to cool to room temperature. Once it has, remove it from the oven, and store in a glass container in a cool dry place until use.



Setup for nitration of benzene-1,3,5-triyltris(chlorane) nonaoxide

Theoretical Preparation 9: 1,3,5-triazido-2,4,6-trinitrobenzene; Nitrazide; TATNB

 1,3,5-triazido-2,4,6-trinitrobenzene	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6N_{12}O_6$
Formula Weight = 336.141
Composition = C(21.44%) N(50.00%) O(28.56%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 336.006376 Da
Nominal Mass = 336 Da
Average Mass = 336.141 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
Estimated 8980	Non-flammable below 100 Celsius. When heated to above 100 Celsius it ignites, and burns slowly emitting toxic fumes.	Stable at room temperature. Non-sensitive to shock, percussion, and friction. Impact test, 1-kilogram drop 7.5 meters. Can also be detonated when hit by a rifle bullet. Requires a blasting cap or detonator for proper initiation. A secondary explosive	Non-reactive.	Colorless to light yellow crystalline mass or powder. Melting point ranges from 156 to 190 Celsius depending on purity. Boiling point: Decomposes above 400 Celsius. Insoluble in water, and most organic solvents. This substance is toxic and known carcinogen, avoid skin contact,

				inhalation, and ingestion of this compound.

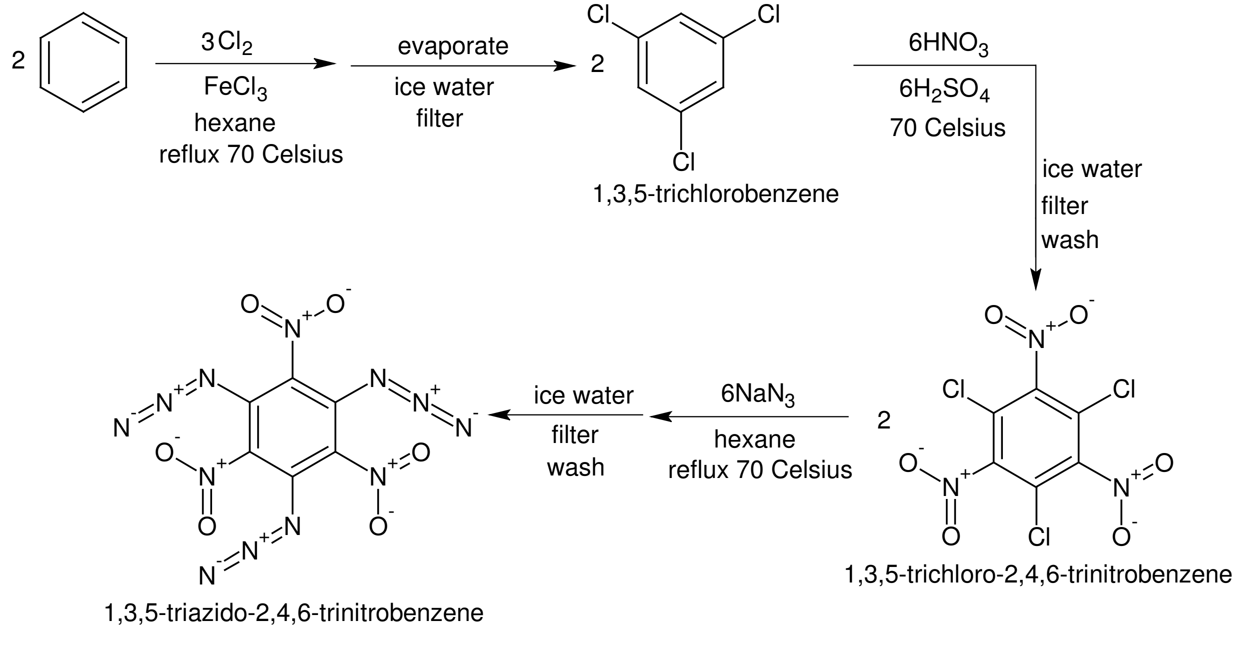
Estimated Compound summary: 1,3,5-triazido-2,4,6-trinitrobenzene is a remarkable explosive with a high degree gas upon detonation making it a powerful explosive for use in all kinds of military operations.1,3,5-triazido-2,4,6-trinitrobenzene has outstanding physical properties and stability, and it is well suitable for use in all sorts of warheads for rockets, mortar shells, artillery shell, missile warheads, and land minds. Because of its high degree of nitrogen and oxygen content, it is a powerful explosive well suitable for use in demolition operations, mining hard rock, and all sorts of blasting and demolition operations. Also, because of its high nitrogen and oxygen content, it makes for powerful charges in anti-ship missiles, and submarine torpedo warheads.

Safety Info: Wear gloves and avoid inhalation of benzene vapors as benzene is a carcinogen. Use proper ventilation and avoid inhalation of chlorine gas which is toxic. Wear gloves and eye protection when handling concentrated nitric and sulfuric acids, which are both very corrosive. Keep concentrated nitric acid away from copper, as toxic fumes will be evolved. Concentrated sulfuric acid is a strong dehydrating agent that will burn and char many organic substances especially wood. Use extreme caution and use proper ventilation when adding concentrated sulfuric acid to concentrated nitric acid as toxic fumes will develop. Extinguish all flames and sources of ignition when handling hexane, which is highly flammable. Wear gloves and avoid skin contact, inhalation, and ingestion of sodium azide, which is a deadly poison.

Materials:

1. 190 grams of dry benzene	5. 700 milliliters of concentrated sulfuric acid
2. 10 grams of anhydrous ferric chloride	6. 2000 milliliters of a 5% sodium carbonate solution
3. 259 grams of dry chlorine gas	7. 2500 milliliters of dry hexane
4. 670 grams of 70% nitric acid	8. 687 grams of sodium azide

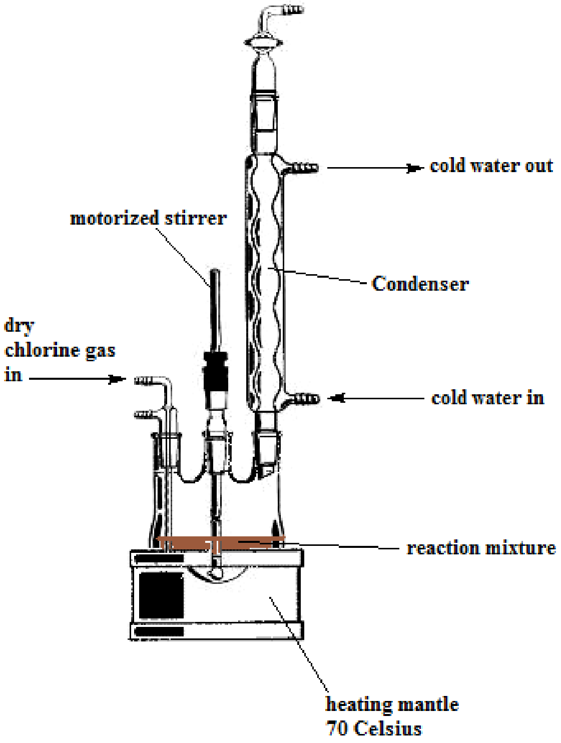
Reaction Equation:



Preparation:

Step 1: Preparation of 1,3,5-trichlorobenzene

Into a suitable apparatus as illustrated below, pour in 190 grams of dry benzene, and then add in 10 grams of anhydrous ferric chloride. Thereafter, add in 500 milliliters of dry hexane, and then assemble the apparatus as illustrated below. Thereafter, heat the contents in the apparatus to 70 Celsius and begin a reflux of the contents by heating to 70 Celsius. When the temperature of the reaction mixture reaches a gentle reflux at 70 Celsius, begin to bubble into the reaction mixture dry chlorine gas, at a flow rate of 500 mg per/minute, while stirring the reaction mixture and maintaining a reflux of 70 Celsius. Continue to bubble in dry chlorine gas until 259 grams of dry chlorine gas has been added. After the addition of the dry chlorine gas, continue to stir and reflux the reaction mixture at 70 Celsius for about 3 hours. After 3 hours, stop stirring and remove the heat source and allow the reaction mixture to cool to room temperature. Now, disassemble the apparatus and then pour the entire reaction mixture into a rotary evaporator, and evaporate-off the hexane under moderate vacuum until dry solid remains. When dry solid remains, stop the evaporation process and then remove the left over dried residue. Then place this left over dried residue into 500 milliliters of ice water, and then stir the entire mixture for 30 minutes to dissolve out the ferric chloride. After stirring for 30 minutes, filter-off the insoluble solid, using suction filtration, and then wash with three 500 milliliter portions of hot water, and then suction dry, and then save the 1,3,5-trichlorobenzene for step 2.



Apparatus for chlorinating benzene.

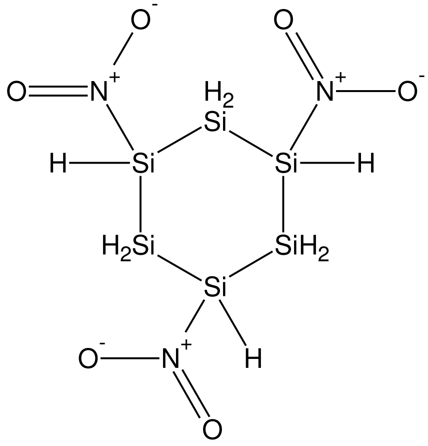
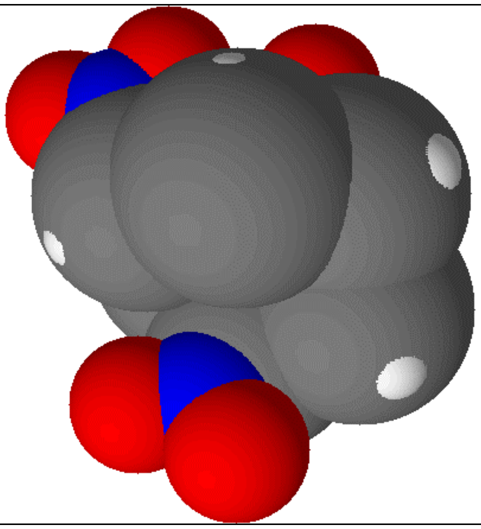
Step 2: Preparation of 1,3,5-trichloro-2,4,6-trinitrobenzene

Into a suitable 3-neck flask, equipped with motorized stirrer, condenser, and thermometer, pour in 670 grams of 70% nitric acid, and then slowly add in, 700 milliliters of concentrated sulfuric acid. Note: use caution when adding the sulfuric acid to the nitric acid. After the addition of the sulfuric acid, allow the acid mixture to cool to around 70 Celsius, then turn on the heating mantle, and set at a temperature of 70 Celsius. When a constant temperature of 70 Celsius has been achieved consistently, slowly add in, in small portions at a time while rapidly stirring the nitrating acid mixture, the 1,3,5-trichlorobenzene obtained from step 1, over a period of 2 hours, while rapidly stirring the reaction mixture and maintaining its temperature around 70 Celsius. After the addition of the 1,3,5-trichlorobenzene, continue to heat and rapidly stir the reaction mixture at 70 Celsius for 6 hours. After 6 hours, remove the heat source and continue to stir the reaction mixture until its temperature reaches room temperature. When the reaction mixtures temperature reaches room temperature, stop stirring, disassemble the apparatus, and then slowly and carefully pour the entire reaction mixture into a large beaker filled 3/4th of the way up the large beaker filled with ice. After the ice has melted, stir the entire mixture for 1 hour, and then add more ice and continue to stir for an additional1 hour. Thereafter, filter-off the precipitated product using suction filtration, and then wash with 2000 milliliters of a 5% sodium carbonate solution, followed by washing with 5000 milliliters of warm water, and then suction dry. Now, remove the washed suction dried product of 1,3,5-trichloro-2,4,6-trinitrobenzene and place in an oven at 100 Celsius for 6 hours. Thereafter remove the dried product, allow it to cool to room temperature, and then save for step 3.

Step 3: Preparation of 1,3,5-triazido-2,4,6-trinitrobenzene

Into a standard reflux apparatus equipped with motorized stirrer, place the dried 1,3,5-trichloro-2,4,6-trinitrobenzene, followed by 2500 milliliters of dry hexane. Thereafter, bring the mixture to a boil, and begin refluxing it. Immediately when the reaction mixture begins to boil, add in small portions at a time, 687 grams of sodium azide over a period of about 1 hour, while rapidly stirring the reaction mixture and maintaining it temperature around 70 to 75 Celsius. Continue to reflux and stir the reaction mixture at 70 to 75 Celsius for 6 hours. After 6 hours, remove the heat source and allow the reaction mixture to cool to room temperature. Once it has, disassemble the apparatus in the usual manner and then pour the entire reaction mixture into a large beaker containing ice water, and then stir the entire mixture for 1 hour. After 1 hour, filter-off the insoluble product using suction filtration, and then wash with three, 500 milliliter portions of cold water, followed by three 500 milliliter portions of hot water, and then suction dry the washed product of 1,3,5-triazido-2,4,6-trinitrobenzene, and then place in an oven and cook at 100 Celsius for 4 hours.

Theoretical Preparation 10: 1,3,5-trinitrohexasilinane; nitrosilane; 2-TNHS

 1,3,5-trinitrohexasilinane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_9N_3O_6Si_6$
Formula Weight = 315.60096
Composition = H(2.87%) N(13.31%) O(30.42%) Si(53.39%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 314.910691 Da
Nominal Mass = 315 Da
Average Mass = 315.601 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7190, estimated through infrared spectroscopy	Highly flammable; will ignite spontaneously in dry air burning with a bright light and	Non-sensitive to impact, percussion, shock, or friction. Stable at room temperature when	Hydrolyzed by water, and moist air. Reacts explosively with halogens especially fluorine.	White amorphous mass or powder. Melting point: 98 Celsius. Boiling point: 189 Celsius.

	emitting toxic fumes of nitrogen oxides.	stored under methylene chloride. Requires blasting cap or detonator for initiation. A secondary explosive.		Decomposes when heated to 210 Celsius. Insoluble in water, and most organic solvents, but is slightly soluble in liquid ammonia

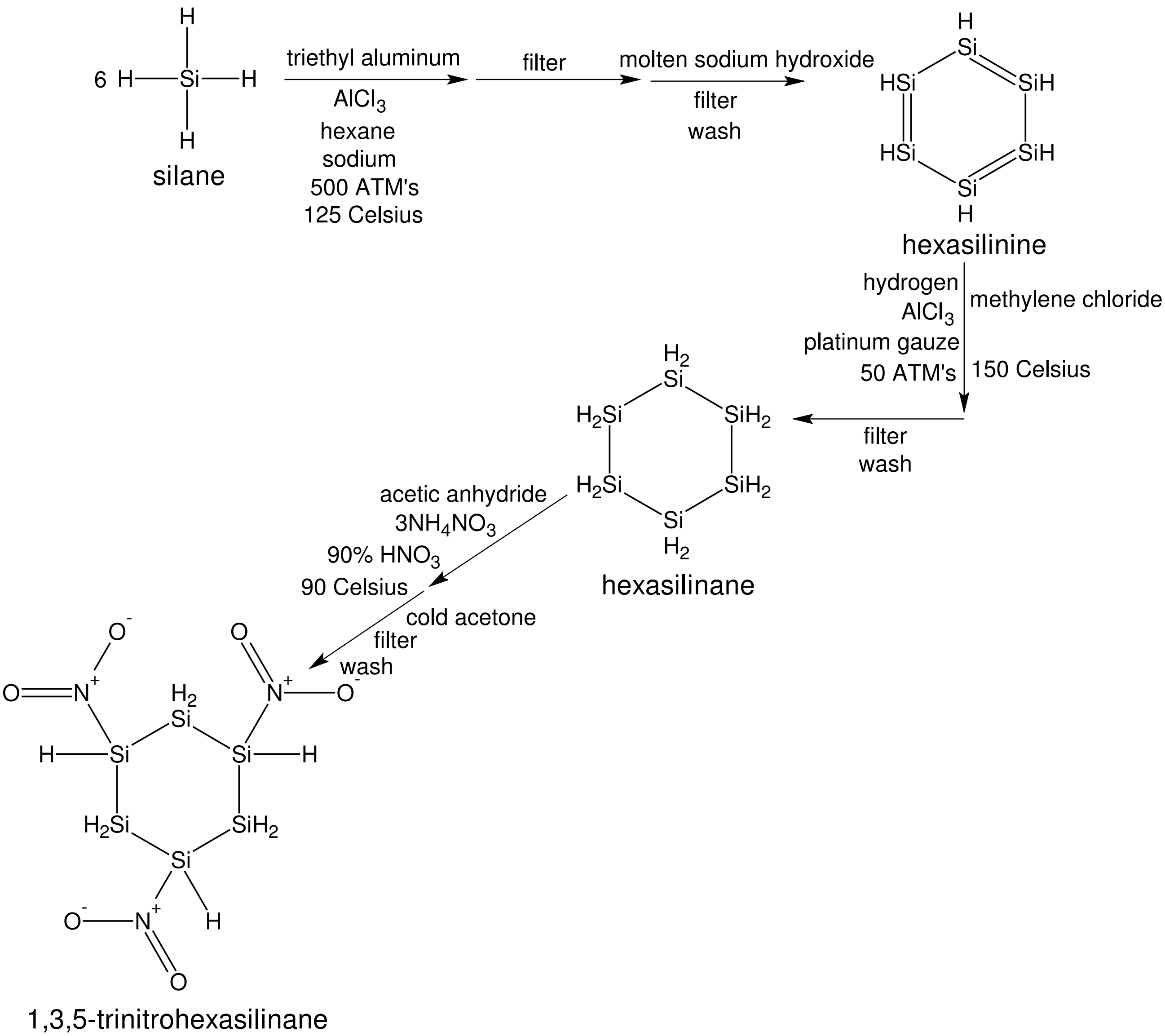
Estimated Compound summary: 1,3,5-trinitrohexasilinane is a very interesting first of its class cyclic silicon based explosive. This unique explosive may not be the first choice for explosives engineers, but it has great shattering power and can be used in demolition operations. However, 1,3,5-trinitrohexasilinane is moisture sensitive hence it is used by mixing it with methylene chloride to form a slurry. This slurry is then packed under pressure into steel containers. 1,3,5-trinitrohexasilinane/methylene chloride slurry explosive can also be used in drop bombs, rocket warheads, and artillery shells, but it has little use in shape charges due to its fluidity. 1,3,5-trinitrohexasilinane shows remarkable use in high performance rocket fuels when cured with epoxy resin, with or without powdered metals.

Safety Info: Extinguish all flames before using hexane, which is highly flammable. Use extreme caution when handling triethyl aluminum, which is spontaneously combustible in air. Use extreme caution when handling metallic sodium, which is very dangerous and explodes in contact with water. Use care when handling anhydrous aluminum chloride, which reacts violently with water. Wear gloves and avoid skin contact when handling sodium hydroxide, which is very corrosive. Extinguish all flames and sources of ignition before using hydrogen gas, which is highly flammable and can explode when mixed with air and ignited. Wear gloves when handling acetic anhydride, which is corrosive and can cause skin irritation. Use proper ventilation, and wear gloves and eye protection when handling 90% nitric acid, which evolves toxic fumes of nitrogen oxides, use care.

Materials:

1. 500 milliliters of dry hexane	8. Dry nitrogen gas from tank
2. 50 grams of triethyl aluminum	9. 4 grams of dry hydrogen gas
3. 98 grams off metallic sodium	10. 700 milliliters of acetic anhydride
4. 25 grams of anhydrous aluminum chloride	11. 145 grams of ammonium nitrate
5. 400 grams of sodium hydroxide	12. 120 milliliters of 90% nitric acid
6. 1350 milliliters of methylene chloride	13. 750 milliliters of chilled acetone
7. 2 grams of platinum gauze	

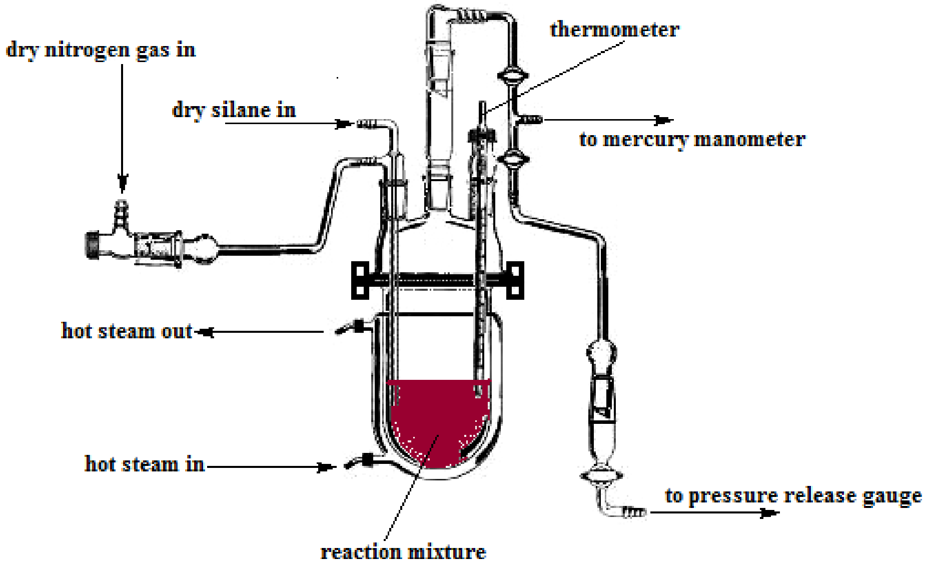
Reaction Equation:



Preparation:

Step 1: Preparation of hexasilinine

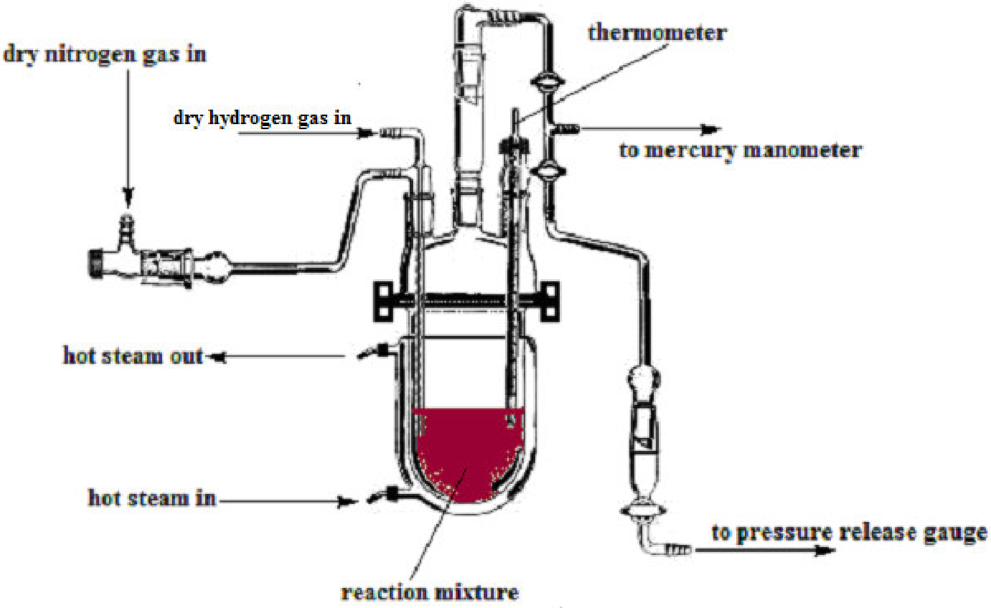
Into a suitable nickel apparatus as illustrated below (note: flush the entire apparatus with dry nitrogen before continuing), place 500 milliliters of dry hexane, followed by 50 grams of triethyl aluminum, followed by 98 grams off metallic sodium, followed by 10 grams of anhydrous aluminum chloride, and then begin to inject steam into the jacket of the reaction vessel. When the temperature inside the reaction vessel reaches around 125 to 130 Celsius, begin to purge the apparatus with dry nitrogen and build up pressure inside the reaction vessel until the pressure gauge (not illustrated) reaches around 500 atmospheres. When it does, begin to bubble dry silane gas into the reaction mixture at a flow rate of 450 mg/per minute, and continue to bubble silane gas into the reaction mixture until 120 grams of silane gas has been added. During the addition of the silane gas, maintain a reaction temperature of 124 to 130 Celsius under a pressure of 500 ATM's at all times. After the addition of the silane gas, continue to heat and maintain pressure of 500 ATM's for 6 hours. After 6 hours, remove the steam cycle, and allow the temperature in the reaction vessel to cool to room temperature. Thereafter, remove the nitrogen purge, and then open the reaction vessel and suction filter the reaction mixture to recover the hexasilinine and sodium hydride. Now, into a suitable beaker place 400 grams of sodium hydroxide, and then heat the sodium hydroxide using a Bunsen burner to melt the sodium hydroxide. Once the sodium hydroxide has melted add in the filtered off product mixture and then stir the molten sodium hydroxide to dissolve the sodium hydride. Continue to stir the molten sodium hydroxide mixture for 1 hour, and then quickly suction filter the molten mixture to recover the insoluble hexasilinine. After filtering-off the hexasilinine wash with three 150-milliliter portions of methylene chloride, and then suction dry the hexasilinine. Thereafter, place the suction dried hexasilinine in an oven and cook at 50 Celsius for 3 hours. After 3 hours remove the heat source and allow the hexasilinine to cool to room temperature, and then save for step 2. Note: hexasilinine is moisture sensitive so store it in a dissecator over concentrated sulfuric acid until use.



All stainless nickel apparatus for polymerization of silane gas.

Step 2. Preparation of hexasilinane

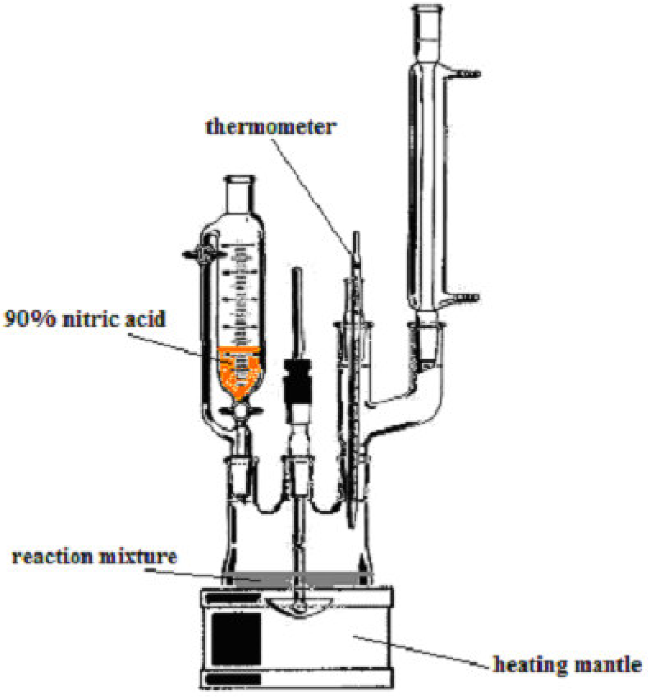
Into an apparatus as illustrated below (purge the apparatus with dry nitrogen before continuing), place 600 milliliters of methylene chloride, followed by 15 grams of anhydrous aluminum chloride, followed by 2 grams of platinum gauze, and then followed by the hexasilinine prepared in step 1. Thereafter, purge the apparatus with dry nitrogen to produce a pressure of 50 ATM's, and begin to pump in steam into the jacket of the reaction vessel to produce a temperature of 150 to 160 Celsius. When the temperature of the reaction mixture reaches 150 to 160 Celsius under a pressure of 50 ATM's, begin to bubble into the reaction mixture dry hydrogen gas at a flow rate of 300 mg per/minute. Continue to heat the reaction mixture at 150 to 160 Celsius under a pressure of 50 ATM's, until 4 grams of dry hydrogen gas has been added. After 4 grams of dry hydrogen gas has been added, continue to heat the reaction mixture at 150 to 160 Celsius under a pressure of 50 ATM's for 4 hours. After 4 hours, remove the steam and allow the temperature of the reaction mixture to cool to room temperature, and then remove the nitrogen purge. Thereafter, open the reaction vessel and pour the entire reaction mixture into a suitable beaker. Thereafter, fish out the platinum gauze using a pair of tweezers, and then filter-off the insoluble hexasilinane using suction filtration. Thereafter, wash the filtered-off hexasilinane with three 150-milliliter portions of fresh methylene chloride, and then suction dry. Thereafter, place this washed filtered-off hexasilinane into an oven and cook at 50 Celsius for 6 hours. After 6 hours, remove the heat source, and allow the hexasilinane to cool to room temperature. Once it has, save it for step 3. Note: hexasilinane is moisture sensitive so store in a dissecator over concentrated sulfuric acid until use.



Glass setup for addition of hydrogen gas under pressure to prepare hexasilinane.

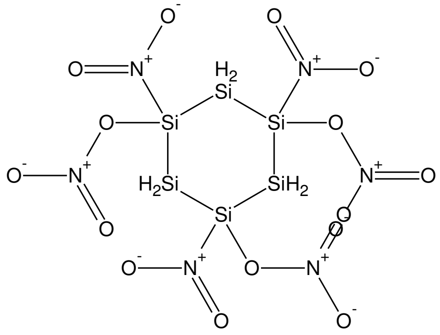
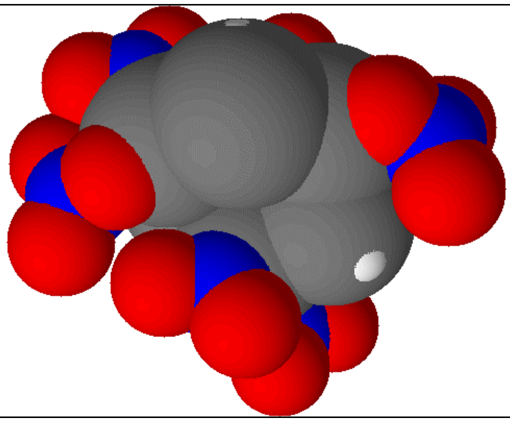
Step 3. Preparation of 1,3,5-trinitrohexasilinane

Into a suitable 3-neck flask, equipped with motorized stirrer, thermometer, and addition funnel, add into the 3-neck flask 700 milliliters of acetic anhydride, followed by 145 grams of ammonium nitrate, and then followed by the hexasilinane prepared in step 2. Thereafter, place 120 milliliters of 90% nitric acid into the addition funnel, and then turn on the heating mantle and heat the contents in the 3-neck flask to 90 Celsius with constant stirring. When the temperature of the reaction mixture reaches 90 Celsius, begin a moderate drip of the 90% nitric acid into the reaction mixture while maintaining the reaction mixtures temperature at 90 Celsius with rapid stirring. After the addition of the 90% nitric acid, continue to stir and heat the reaction mixture at 90 Celsius for about 1 hour. After 1 hour, remove the heat source and allow the reaction mixtures temperature to cool to room temperature. Once it has, disassemble the apparatus, and then pour the entire reaction mixture into a large beaker filled with 750 milliliters of chilled acetone (cooled in a freezer), and then stir the entire mixture for about 1 hour. Thereafter, filter-off the insoluble 1,3,5-trinitrohexasilinane using suction filtration, and then wash the filtered-off product with three 150-milliliter portions of methylene chloride, and then suction dry. Thereafter, place the washed product into an oven and cook at 70 Celsius for 4 hours. After 4 hours, remove the heat source, and allow the 1,3,5-trinitrohexasilinane to cool to room temperature. Note: store the 1,3,5-trinitrohexasilinane in a dissecator over concentrated sulfuric acid until use.



Set-up for nitration of hexasilinane.

Theoretical Preparation 11: 1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium; TNNHS; Si-135

 1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_6N_6O_{15}Si_6$
Formula Weight = 498.59184
Composition = H(1.21%) N(16.85%) O(48.13%) Si(33.80%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 497.85067 Da
Nominal Mass = 498 Da

Average Mass = 498.5918 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
8120; estimated using infrared spectroscopy	Flammable; but requires ignition from a flame. Does not ignite when heated. Burns with a reddish flame emitting toxic fumes of nitrogen oxides.	Non-sensitive. Stable to percussion, shock, and friction. Requires blasting cap or detonator for initiation. A secondary explosive.	Relatively non-reactive and shows remarkable stability. Stable in moist air, and only slowly reacts with water. Shows little reaction with the halogens with the exception of fluorine; explodes in contact with fluorine.	Colorless, transparent cubic like crystals, or white powder. Melting point: 123 Celsius; Boiling point: Decomposes at 290 Celsius, but can be distilled under high vacuum at 180 Celsius. Insoluble in water, and most organic solvents. Shows moderate solubility in liquid ammonia and liquid sulfur dioxide.

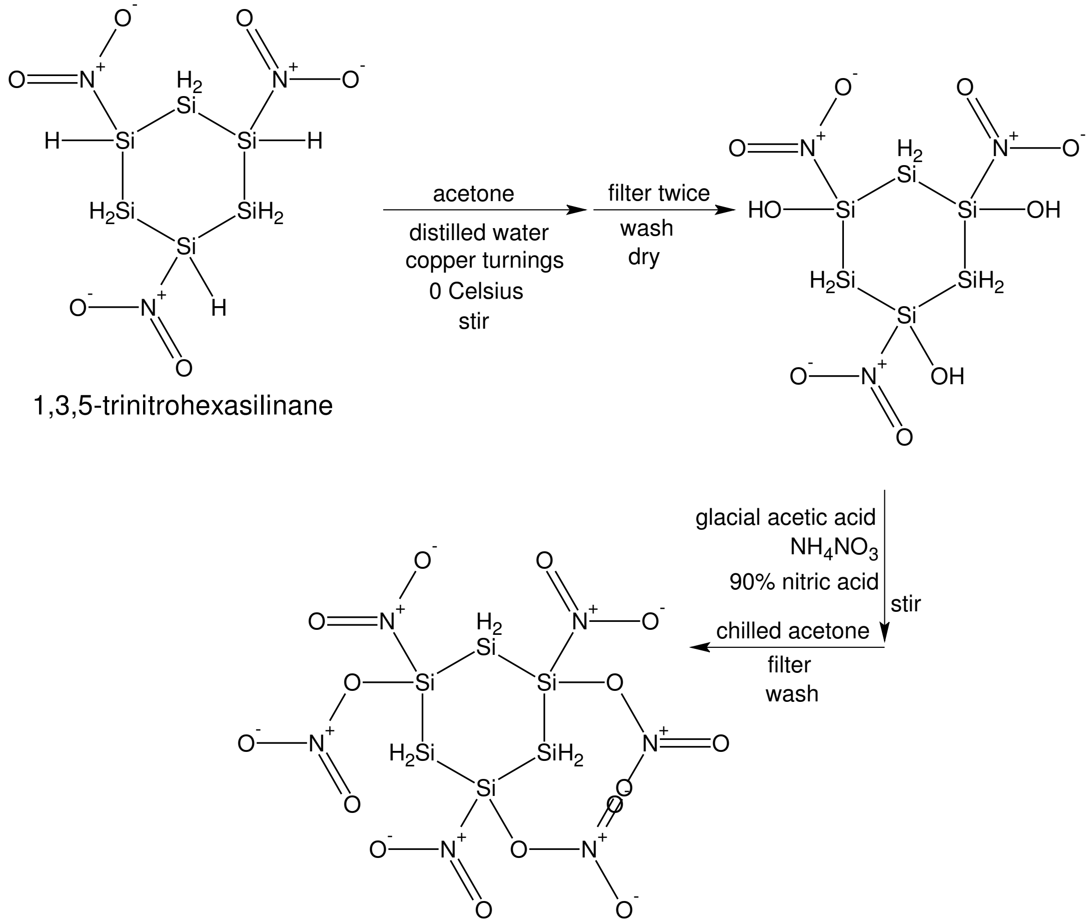
Compound summary: 1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium is a remarkably stable and fascinating silicon based high explosive with tremendous energy. It is much more stable then its predecessor 1,3,5-trinitrohexasilinane and with its relatively moderate melting point, it can be melted and casted into shape charges for a wide variety of penetrating munitions including anti-tank weapons, anti-ship weapons, and submarine torpedoes. It can also be used in forming demolition charges, explosive compositions admixed with other explosives, or in the manufacture of blasting charges for mining hard or soft rock. It can also be used in high performance rocket propellants when admixed with powdered metals and cured with epoxy resins.

Safety Info: Extinguish all flames and sources of ignition when handling acetone, which is highly flammable. Wear gloves when handling glacial acetic acid, which can cause skin irritation. Use proper ventilation, wear gloves and eye protection when handling 90% nitric acid, and avoid inhalation of the toxic fumes, use extreme care.

Materials:

1. 500 milliliters of acetone	6. 800 milliliters of glacial acetic acid
2. 50 grams of copper turnings	7. 90 grams of ammonium nitrate
3. 145 grams of 1,3,5-trinitrohexasilinane (prepared in theoretical preparation 10, step 3)	8. 95 grams of 90% nitric acid
4. 24 milliliters of distilled water	9. 750 milliliters of chilled acetone
5. 900 milliliters of methylene chloride	

Reaction Equation:



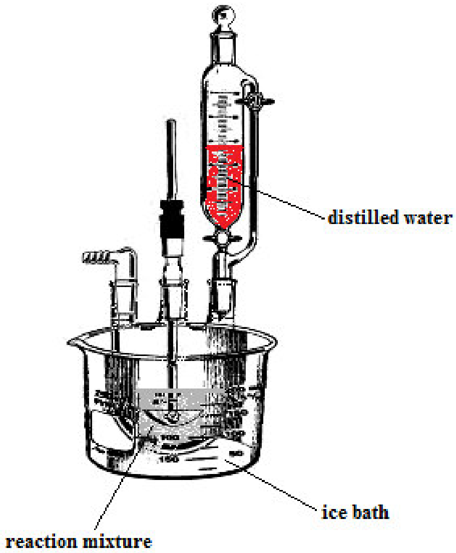
1,3,5-trinitrohexasilinane

1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium

Preparation:

Step 1: Preparation of 1,3,5-trihydroxy-1,3,5-trinitrohexasilinane-1,3,5-triium

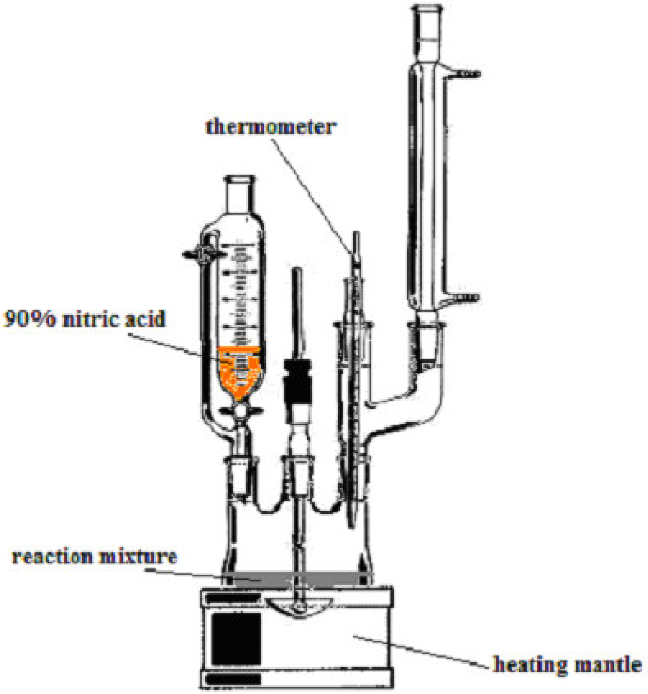
Into a setup as illustrated below, add in 500 milliliters of acetone, followed by 50 grams of copper turnings, and then followed by 145 grams of 1,3,5-trinitrohexasilinane (prepared in theoretical preparation 10, step 3). Thereafter assemble the apparatus as illustrated below and then place 24 milliliters of distilled water into the addition funnel. Thereafter, chill the apparatus in an ice bath, and when the temperature of the reaction mixture reaches 0 Celsius, begin a moderate drip of the distilled water into the acetone reaction mixture over a period of about 1 hour, while maintaining the reacting mixtures temperature around 0 Celsius, with constant stirring. After the addition of the distilled water, continue to stir the reaction mixture at 0 Celsius for 2 hours. After 2 hours, remove the ice bath and allow the reaction mixture to warm to room temperature, and then continue to blend the reaction mixture for 4 hours. After 4 hours, suction filter the reaction mixture using a Buchner funnel with no filter paper, so that the insoluble finely divided product can pass through, but the copper turnings will be captured. Once the copper turnings have been removed, re-filter the filtered reaction mixture using suction filtration and fine grade filter paper to collect the insoluble desired product of 1,3,5-trihydroxy-1,3,5-trinitrohexasilinane-1,3,5-triium. Thereafter, wash with three 150-milliliter portions of methylene chloride, and then suction dry the 1,3,5-trihydroxy-1,3,5-trinitrohexasilinane-1,3,5-triium. Finally place this dried product into an oven, and cook at 50 Celsius for 1 hour. After 1 hour, remove the heat source, and then allow the product to cool to room temperature. Thereafter save for step 2.



Setup for addition of water.

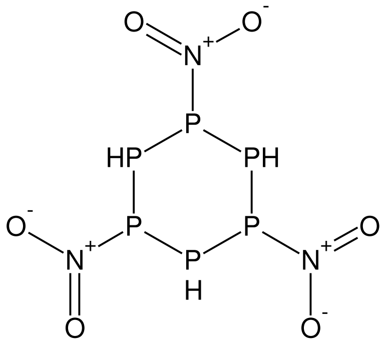
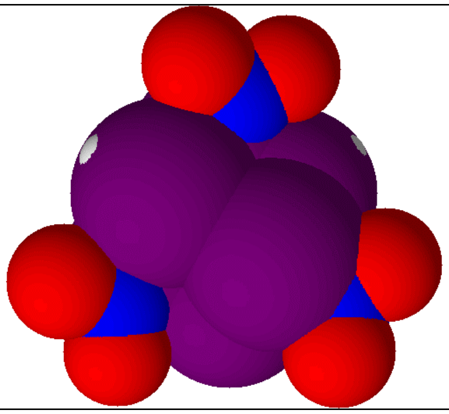
Step 2. Preparation of 1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium

Into a suitable 3-neck flask, equipped with motorized stirrer, thermometer, and addition funnel, add into the 3-neck flask 800 milliliters of glacial acetic acid, followed by 90 grams of ammonium nitrate, and then followed by the 1,3,5-trihydroxy-1,3,5-trinitrohexasilinane-1,3,5-triium prepared in step 1. Thereafter, place 95 grams of 90% nitric acid into the addition funnel, and then turn on the heating mantle and heat the contents in the 3-neck flask to 90 Celsius with constant stirring. When the temperature of the reaction mixture reaches 90 Celsius, begin a moderate drip of the 90% nitric acid into the reaction mixture while maintaining the reaction mixtures temperature at 90 Celsius with rapid stirring. After the addition of the 90% nitric acid, continue to stir and heat the reaction mixture at 90 Celsius for about 1 hour. After 1 hour, remove the heat source and allow the reaction mixtures temperature to cool to room temperature. Once it has, disassemble the apparatus, and then pour the entire reaction mixture into a large beaker filled with 750 milliliters of chilled acetone (cooled in a freezer), and then stir the entire mixture for about 1 hour. Thereafter, filter-off the insoluble 1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium using suction filtration, and then wash the filtered-off product with three 150-milliliter portions of methylene chloride, and then suction dry. Thereafter, place the washed product into an oven and cook at 70 Celsius for 4 hours. After 4 hours, remove the heat source, and allow the 1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium to cool to room temperature.



Setup for the preparation of 1,3,5-trinitro-1,3,5-tris(nitrooxy)hexasilinane-1,3,5-triium

Theoretical Preparation 12: 1,3,5-trinitrohexaphosphinane; TNHP; High Explosive Phosphorus

 1,3,5-trinitrohexaphosphinane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_3N_3O_6P_6$
Formula Weight = 326.882892
Composition = H(0.93%) N(12.85%) O(29.37%) P(56.85%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 326.844751 Da
Nominal Mass = 327 Da

Average Mass = 326.8829 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6170	Flammable solid. Burns with a yellow flame producing white fumes of phosphorus oxides. Not spontaneously combustible, but will spontaneously ignite in air when heated to 300 Celsius.	Non-sensitive. Stable to heat, percussion, shock, and friction when pure. When admixed with powdered metals, it ignites and burns rapidly upon friction (hence could be used to make matches). Requires blasting cap or detonator for initiation. A secondary explosive	Slowly reacts with water and moisture. Keep stored in an air tight, watertight container, preferably a dissecator over concentrated sulfuric acid to protect from moisture.	Yellow amorphous solid. Melting point: 189 Celsius when pure; commercial grades most likely would have a melting point of 210 Celsius. Boiling point: Decomposes when heated above 450 Celsius. Can be distilled in a stream of dry hydrogen chloride gas under reduced pressure at 190 Celsius. Insoluble in water, which it is slowly hydrolyzed, and insoluble in most organic solvents. Limited solubility in liquid ammonia.

Estimated Compound summary: In the great light of the silicon based high explosives, 1,3,5-trinitrohexaphosphinane is an interesting phosphorus based explosive with interesting properties. Although this explosive is rather exotic and probably not of much use or interest, it shows remarkable properties as a high explosive and can be used when dead pressed into bomb casings, and shell casings under 2500 psi. Although not of much commercial or military value as a high explosive, it does show remarkable interest in manufacturing high performance gun propellants, high performance rocket propellants, and in the manufacturing pyrotechnics compositions including matches, flares, and fireworks. 1,3,5-trinitrohexaphosphinane ignites rapidly and burns with great brilliance when admixed with powdered aluminum; it burns with a gorgeous deep blue flame when mixed with copper dust, and it burns rapidly when mixed with reducing agents and ignited; therefore, its use in manufacturing rocket fuels is clearly evident when admixed with reducing agents and cured with epoxy.

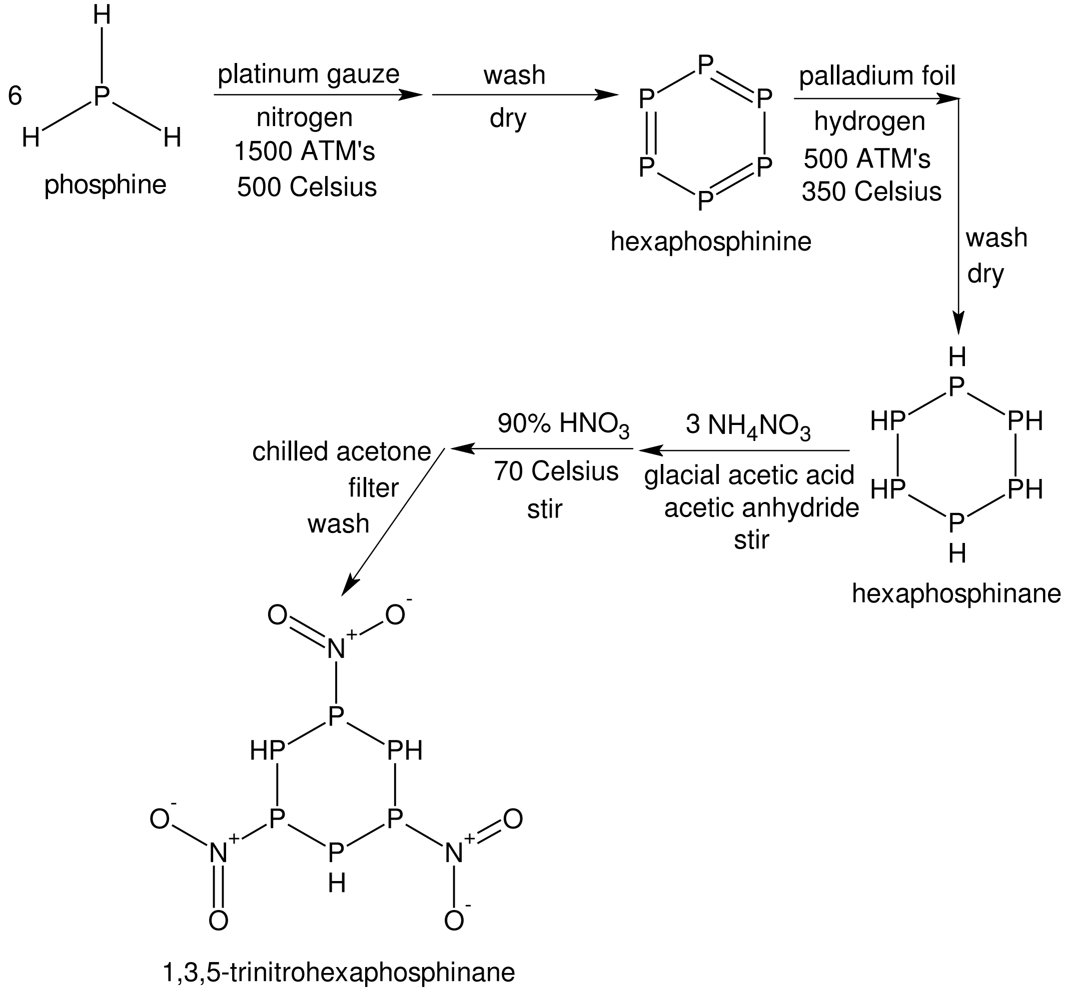
Safety Info: Use extreme caution when handling phosphine gas, which is spontaneously combustible in air, and may explode in contact with oxygen, also use proper ventilation when handling phosphine gas, which is toxic. Wear gloves when handling glacial acetic acid and acetic anhydride, which are corrosive and can cause skin irritation. Use proper ventilation, and wear gloves and eye protection, and avoid inhalation of the fumes when handling 90% nitric acid, which evolves toxic fumes of nitrogen oxides. Acetone is highly flammable so extinguish all sources of ignition prior to using.

Materials:

1. 10 grams of platinum gauze	8. 400 milliliters of glacial acetic acid
2. Nitrogen gas from tank	9. 300 milliliters of acetic anhydride
3. 190 grams of phosphine gas	10. 105 milliliters of 90% nitric acid
4. 1500 milliliters of ice water	11. 750 milliliters pre-chilled acetone (chilled in a freezer prior to use)
5. 50 grams of palladium foil	12. 450 milliliters of dry acetone

6. 450 milliliters of methylene chloride	13. 15 grams of hydrogen gas
7. 188 grams of ammonium nitrate	

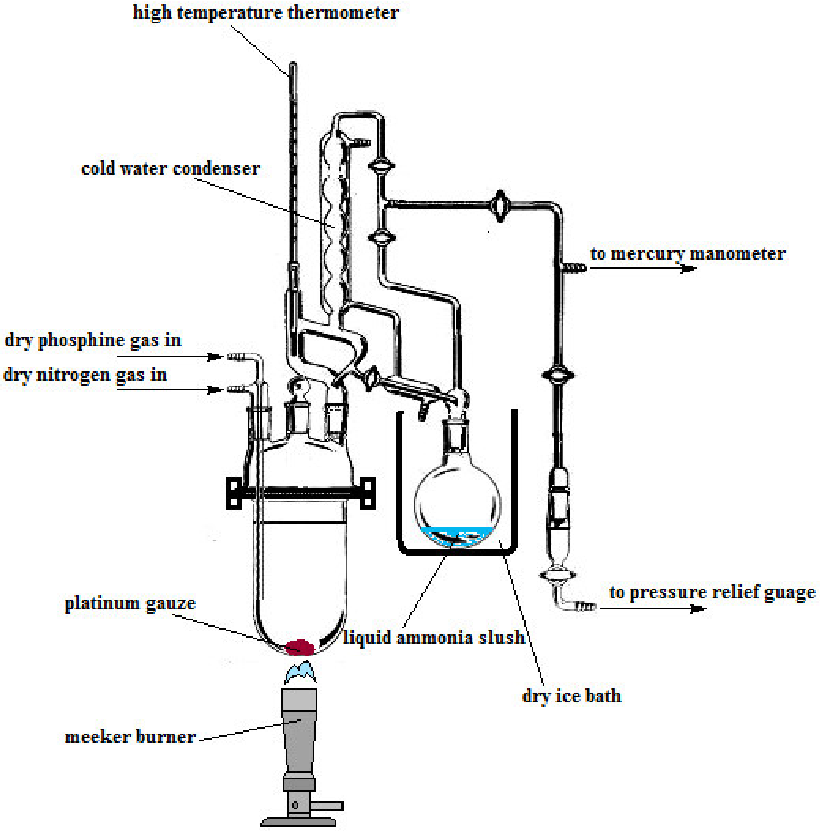
Reaction Equation:



Preparation:

Step 1: Preparation of hexaphosphinine

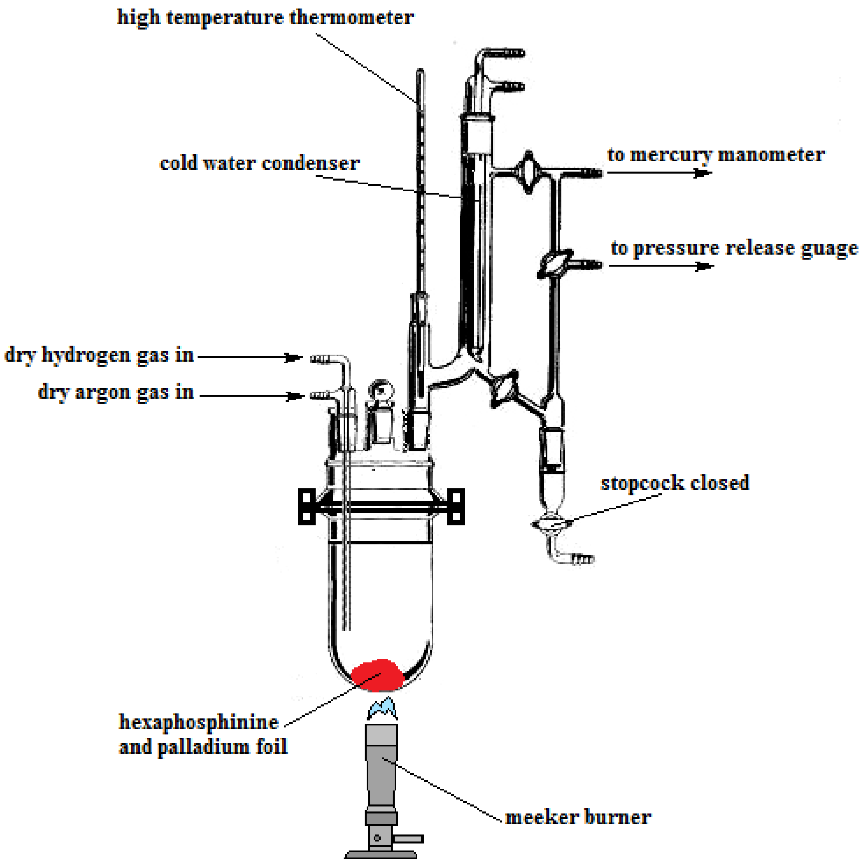
Into an advanced steel apparatus as illustrated below, place 10 grams of platinum gauze, and then seal the apparatus. Thereafter begin a nitrogen purge, and create a pressure of 1500 ATM's, and turn on the meeker burner and heat the inside of the reaction vessel to 500 Celsius. When the pressure inside the reaction vessel reaches 1500 ATM's, and a temperature of 500 Celsius, begin to pass a dry current of phosphine gas into the reaction vessel at a flow rate of 50 mg per/minute. Continue to pass in dry phosphine gas into the reaction vessel until 190 grams of phosphine has been passed into the reaction vessel. After 190 grams of phosphine gas has been passed into the reaction vessel continue to heat and maintain a temperature and pressure of 500 Celsius at 1500 ATM's for about 4 hours. After 4 hours, remove the heat source, release the pressure gauge to bring the pressure inside the reaction vessel back to atmospheric pressure and allow the contents in the reaction vessel to cool to room temperature. Thereafter, disassemble the apparatus, and then scrap the desired hexaphosphinine from the sides of the reaction vessel, and then recover the platinum gauze. Then put the scrapped off hexaphosphinine into a Buchner funnel with slow flow rate filter paper, and then wash the hexaphosphinine with three-500 milliliter portions of ice water using suction filtration, and then after the washings, suction dry the desired hexaphosphinine, and then save for step 2.



Stainless steel apparatus for preparing hexaphosphinine.

Step 2 Preparation of hexaphosphinane

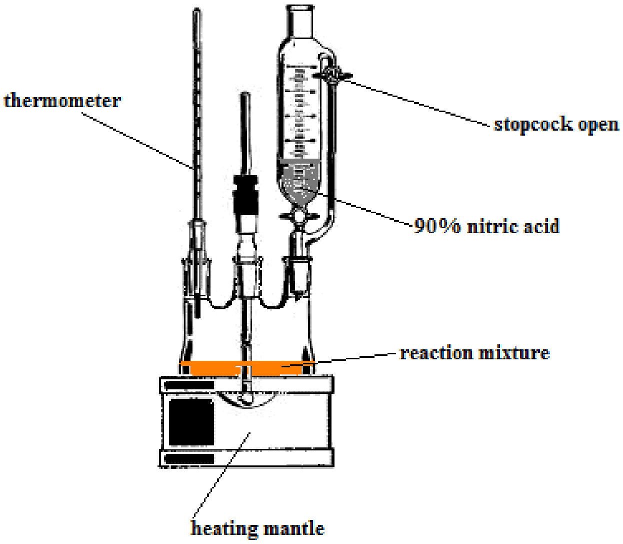
Into an apparatus as described below, add in the hexaphosphinine obtained from step 1, and then add in 50 grams of palladium foil, and then begin a argon gas purge by flushing the apparatus with argon, and then controlling the setting on the pressure gauge (not illustrated), begin a pressure of 500 ATM's, and then turn on the meeker burner and let the temperature in the reaction vessel to 350 Celsius. When the pressure and heat in the reaction vessel reaches 500 ATM's, at a temperature of 350 Celsius begin to pump in dry hydrogen gas at a flow rate 50 mg per/minute. Continue to pump in dry hydrogen gas until 15 grams of hydrogen gas has been added. After the hydrogen gas has been added, continue to heat the contents in the reaction vessel to 350 Celsius under a pressure of 500 ATM's for 3 hours. After 3 hours, remove the heat source, and release the pressure gauge to bring the pressure inside the reaction vessel back to atmospheric pressure. When the contents in the reaction vessel has reached room temperature, open up and disassemble the apparatus, and then scrape the desired hexaphosphinane from the sides of the reaction vessel, and then place this scraped material into a Buchner funnel fitted with a slow rate filter paper (note: then recover the palladium foil, wash with hot water and save for another run or different procedure). Now, to the desired hexaphosphinane in the Buchner funnel, wash this material with three 150-milliliter portions of methylene chloride using suction filtration, and then suction dry the washed product. Finally, place the washed hexaphosphinane into an oven, and bake at 60 Celsius for 6 hours. After 6 hours, turn off the heat and allow the dried hexaphosphinane to cool to room temperature, and then save for step 3.



All nickel apparatus for addition of hydrogen.

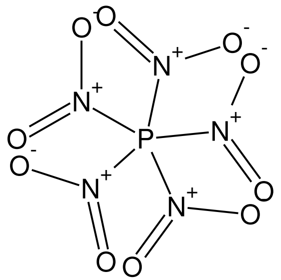
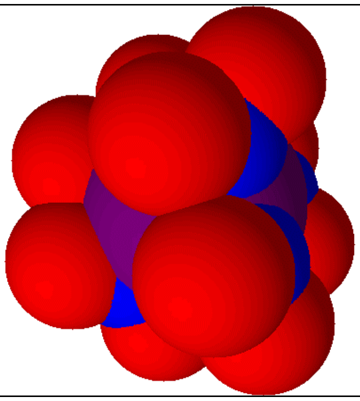
Step 3: Preparation of 1,3,5-trinitrohexaphosphinane

Into a suitable 3-neck flask, equipped with addition funnel, thermometer, and heating mantle, place 188 grams of ammonium nitrate, followed by 400 milliliters of glacial acetic acid, followed by 300 milliliters of acetic anhydride, and then followed by the hexaphosphinane prepared in step 2. Thereafter, pour 105 milliliters of 90% nitric acid into the addition funnel, and then turn on the heating mantle, and heat the ingredients into the 3-neck flask to 70 Celsius with heavy stirring. When the temperature of the reaction mixture reaches 70 Celsius, begin to drip slowly, the 90% nitric acid over a period of 2 hours, while rapidly stirring the reaction mixture and maintaining its temperature around 70 Celsius. Once all the 90% nitric acid has been added, continue to stir and heat the reaction mixtures temperature at 70 Celsius for 4 hours. After 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Once the reaction mixture has cooled to room temperature, pour the entire reaction mixture into a large beaker containing 750 milliliters pre-chilled acetone (chilled in a freezer prior to use), and then stir the cold mixture for 1 hour. Thereafter, filter-off the insoluble product of 1,3,5-trinitrohexaphosphinane using suction filtration, and then was the filtered-off product with three-150 milliliters of dry acetone, and then suction dry the washed product. Finally, place the washed suction dried product of 1,3,5-trinitrohexaphosphinane into an oven and bake at 80 Celsius for 4 hours. After 4 hours, turn off the heat, and then allow the 1,3,5-trinitrohexaphosphinane to cool to room temperature. Note: store the product in a dissecator over concentrated sulfuric acid until use.



Setup for addition of nitric acid.

Theoretical Preparation 13: pentanitro-λ^5-phosphane; 5-PNP

 pentanitro-λ^5-phosphane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $N_5O_{10}P$
Formula Weight = 261.001262
Composition = N(26.83%) O(61.30%) P(11.87%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 260.938277 Da
Nominal Mass = 261 Da
Average Mass = 261.0013 Da

Molecular Information:

Detonation velocity (estimated by	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

monoisotopic mass and index of refraction):				
7890; estimated using infrared spectroscopy	Generally non-flammable, but will ignite spontaneously when heated above 400 Celsius in air.	Non-sensitive to shock, percussion, friction, and pressure. Requires blasting cap or detonator for initiation. A secondary explosive.	Generally non reactive, but slowly hydrolysizes in moist air and by water. Will be completely and rapidly decomposed when added to boiling water, may also explode when added to boiling water.	Shiny metallic looking reddish yellow crystals or powder. Melting point: 189 Celsius; boiling point: decomposes when heated to above 400 Celsius. Insoluble in water, and most organic solvents. Moderately soluble in liquid ammonia, and liquid sulfur dioxide.

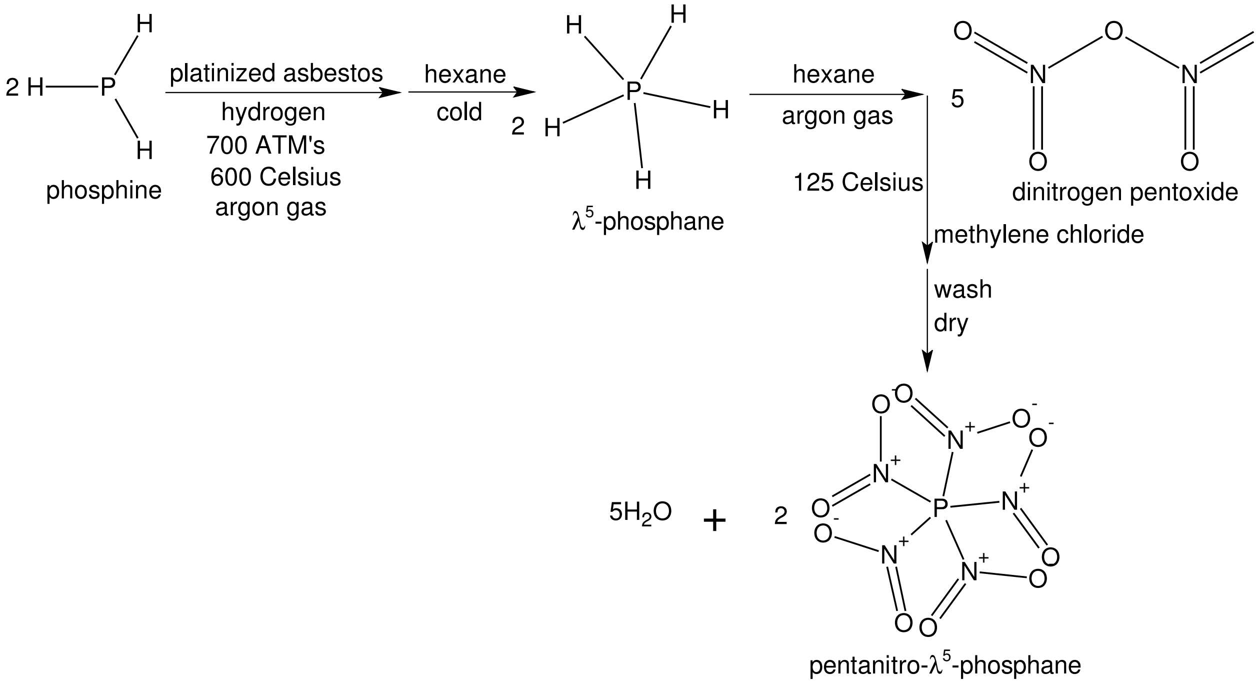
Estimated Compound summary: pentanitro-λ^5-phosphane is a first of its kind nitrated phosphorus-containing compound that has high explosive properties. It shows remarkable stability owing that its chiral phosphorus center is in the 5th oxidation state. Now, it is highly unlikely that this compound will become common place in the world of explosives, but it's interesting nature warrants further investigation. Its relatively high melting point makes it suitable for use as a high explosive in missile warheads, high velocity projectiles, and nuclear weapons. pentanitro-λ^5-phosphane may also be used in making a variety of pyrotechnic compositions as it burns with great brilliance when admixed with powdered metals and reducing agents and ignited. pentanitro-λ^5-phosphane may also be used in making high performance gun propellants, and high performance rocket propellants in the usual means.

Safety Info: Use extreme caution when handling phosphine gas, which is spontaneously combustible; use maximum ventilation, and wear face respirator, as phosphine gas is highly toxic. Extinguish all sources of ignition, including electric arcs when handling hydrogen gas, which is highly flammable and explosive. Extinguish all flames and sources of ignition when handling hexane and acetone, both of which are highly flammable. When dissolved in methylene chloride, dinitrogen pentoxide is relatively safe to store, handle, and use, but avoid contact with many organic materials and water as fire may result, and possible explosions may happen.

Materials:

1. 50 grams of platinized asbestos	5. 570 milliliters of dry hexane
2. Dry argon gas from tank	6. 992 grams of dinitrogen pentoxide
3. 125 grams of dry phosphine gas	7. 2450 milliliters of dry methylene chloride
4. 10 grams of dry hydrogen gas	8. 500 milliliters of pure dry pre-chilled dry acetone

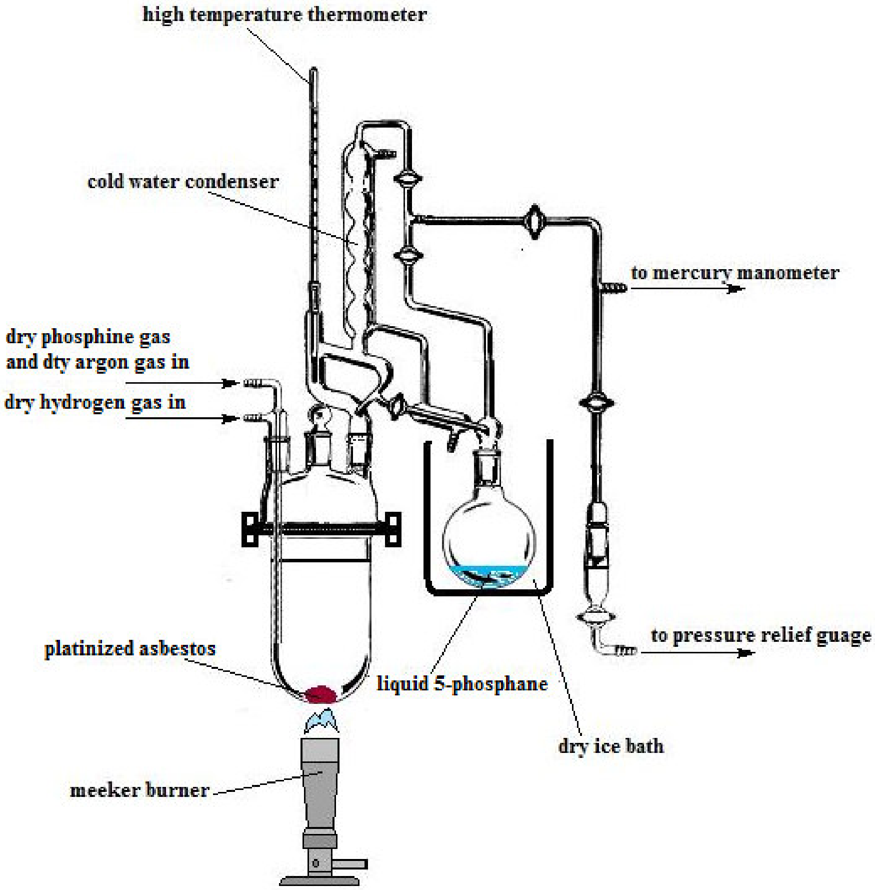
Reaction Equation:



Preparation:

Step 1: Preparation of λ^5-phosphane

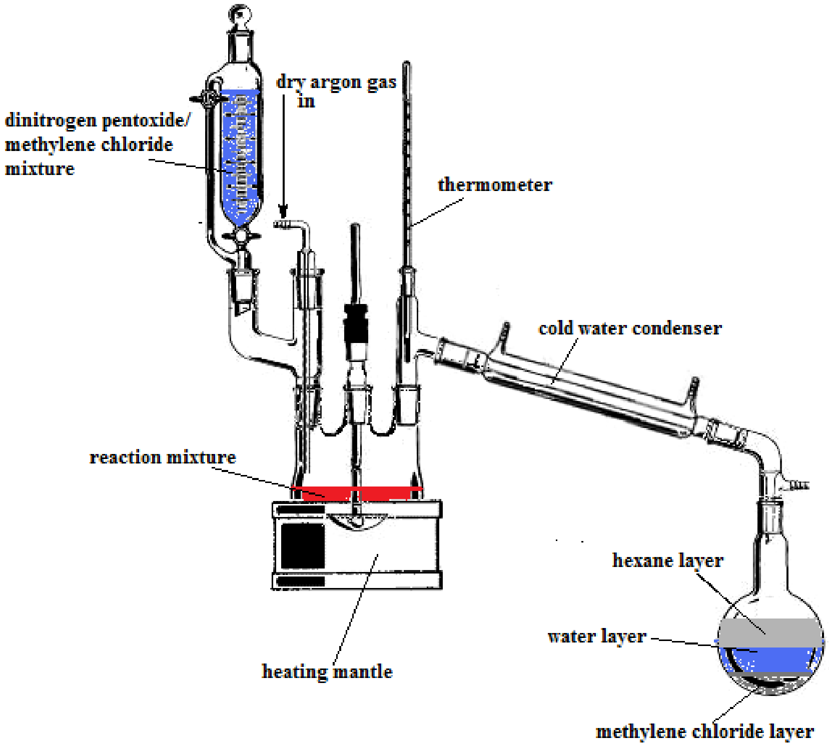
Into an advanced nickel apparatus as illustrated below place 50 grams of platinized asbestos, and then flush and purge the apparatus with dry argon gas prior to pumping in the phosphine or hydrogen gases. Purge the apparatus with argon gas until the internal pressure inside the reaction vessel reaches 700 ATM’s. Then turn on the meeker burner, and heat the reaction vessel to 600 Celsius. When the temperature and pressure inside the reaction vessel is 700 ATM’s at 600 Celsius, begin to pass in dry hydrogen gas and dry phosphine gas at a flow rate of 30 mg per/minute per gas. Continue to pump in dry phosphine and dry hydrogen gas into the reaction vessel until 125 grams of dry phosphine gas has been added, and 10 grams of dry hydrogen gas has been added. After the addition of the dry phosphine and hydrogen gases have been added, continue to heat the reaction vessel at 600 Celsius under a pressure of 700 ATM’s for 1 hour. After 1 hour, remove the heat source, and then release the pressure gauge to reduce the pressure in the reaction vessel to atmospheric pressure. Thereafter, disassemble the apparatus, and immediately remove the flask containing the liquefied λ^5-phosphane slush, and then take this flask, and pour into it, 570 milliliters of dry hexane, and then keep for step 2.



Similar all nickel apparatus for preparation of λ^5-phosphane.

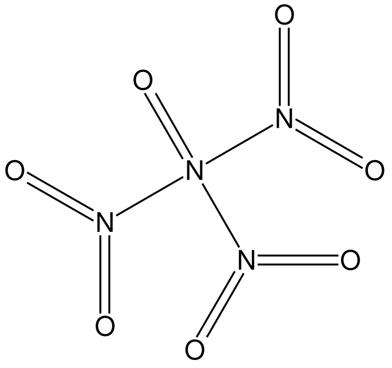
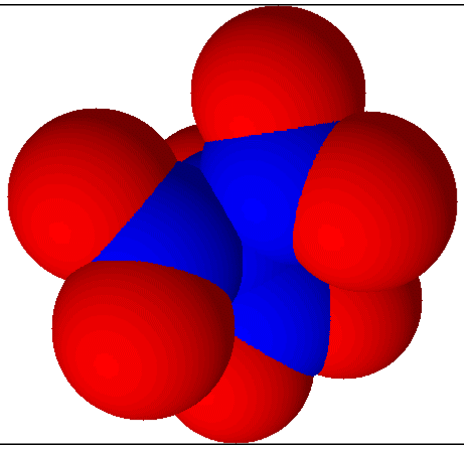
Step 2: Preparation of pentanitro-λ^5-phosphane

To the flask containing the λ^5-phosphane slush/hexane mixture obtained in step 1, mix the ingredients thoroughly for 10 minutes, and then pour all the contents of the flask into a 3-neck flask and assemble the apparatus as illustrated below. Thereafter, place into the addition funnel, 1/3rd of a total mixture of dinitrogen pentoxide dissolved in methylene chloride (prepared by dissolving 992 grams of dinitrogen pentoxide into 2000 milliliters of dry methylene chloride. Note: dinitrogen pentoxide is made by dehydrating concentrated nitric acid with phosphorus pentoxide in the presence of methylene chloride. The dinitrogen pentoxide will dissolve in the methylene chloride, and the water layer can be removed by using a separatory funnel). Thereafter, purge the entire apparatus with dry argon gas, and then heat the contents of the 3-neck flask to 125 Celsius with rapid stirring, and when the temperature of the reaction mixture reaches 125 Celsius, begin a moderate drip of the dinitrogen pentoxide/methylene chloride mixture from the addition funnel over a period of 30 minutes while rapidly stirring the reaction mixture and maintaining its temperature at 125 Celsius. Once all the dinitrogen pentoxide/methylene chloride mixture has been added, pour in another 1/3rd of fresh dinitrogen pentoxide/methylene chloride mixture, and then begin to immediately thereafter, continue a moderate drip of this nitrating mixture to the reaction flask over a period of about 35 minutes, while maintaining the reaction mixtures temperature at 125 Celsius with rapid stirring. Once all the dinitrogen pentoxide/methylene chloride has been added, pour into the addition funnel, the last 1/3rd of the dinitrogen pentoxide/methylene chloride mixture, and then continue a moderate drip of the dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while maintaining the temperature of the reaction mixture at 125 Celsius with rapid stirring. After all the dinitrogen pentoxide/methylene chloride mixture has been added to the reaction mixture, continue to heat the reaction mixture at 125 Celsius with rapid stirring for 3 hours. After 3 hours, remove the heat source, and then allow the contents in the reaction flask to cool room temperature while maintaining rapid stirring during the entire cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire remaining contents in the 3-neck reaction flask into a large beaker filled with 500 milliliters of pure dry pre-chilled dry acetone (this chilled acetone can be chilled in a freezer prior to use). Thereafter, stir the entire mixture for 1 hour, and then filter off the insoluble product using suction filtration, and then wash with three 150-milliliter portions of fresh methylene chloride, and then suction dry the washed product. Finally, place this washed dried product of pentanitro-λ^5-phosphane into an oven and bake at 60 Celsius for 6 hours. After 6 hours, turn off the heat, and then allow the pentanitro-λ^5-phosphane to cool to room temperature; thereafter, store in a dessicator over concentrated sulfuric acid until use.



Setup for nitration of λ^5-phosphane.

Theoretical Preparation 14: trinitroamine oxide; TNAOX; NITROXIDE

 trinitroamine oxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = N_4O_7
Formula Weight = 168.0226
Composition = N(33.34%) O(66.66%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available

Monoisotopic Mass = 167.976698 Da
Nominal Mass = 168 Da
Average Mass = 168.0226 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7100, estimated, similar to nitroglycerin	Non-Flammable. Will ignite spontaneously in moist air if admixed with powdered red phosphorus. Will explode if admixed with powdered metals and ignite by heat, spark, or flame.	Moderately sensitive to shock and percussion, but non-sensitive to bumping, grounding, or friction. Will detonate if a small drop is struck by a hammer. Requires blasting cap or detonator for proper initiation. A secondary explosive.	Generally non-reactive, but it reacts with all the halogens, some with explosive violence. The compound can be reduced with finely divided tin and concentrated hydrochloric acid.	Colorless, syrupy liquid resembling nitroglycerine in nature. Boiling point: 126 Celsius under reduced pressure. Melting point: -78 Celsius. Detonates when heated to 154 Celsius at atmospheric pressure. Soluble in many organic solvents including acetone, alcohols, methylene chloride, hexane, and DMSO, but insoluble in water. Warning! Keep away from powdered metals, chlorine, and the halogens. Also, avoid inhalation, ingestion, or skin contact as this compound is toxic and can produce severe migraines.

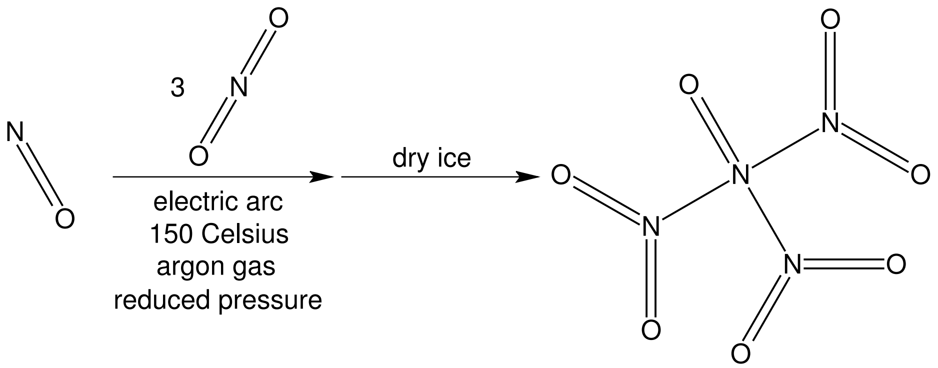
Estimated Compound summary: trinitroamine oxide is a new and experimental liquid explosive very similar to nitroglycerine. trinitroamine oxide is designed to replace the aging explosive nitroglycerine for general blasting operations including blasting soft and hard rock, mining operations, bore-holing, and for use in demolition operations. Like its predecessor, nitroglycerine, trinitroamine oxide is a liquid explosive with very similar characteristics as nitroglycerine, but with much greater stability for long-term storage and handling. Like nitroglycerine, trinitroamine oxide can be absorbed by a number of materials including diatomaceous earth, clays, aluminum oxide powder, and charcoal powder; these materials can be used to absorb trinitroamine oxide up to 70% by weight, and hence used as a safe, non-sensitive explosive compositions for multiple uses. trinitroamine oxide can also be used as a replacement for nitroglycerine in double based high performance gun propellants and rockets fuels especially due to its high gas ratio upon ignition. Unlike nitroglycerine, trinitroamine oxide is completely converted into gases upon ignition and detonation, making it well suitable as a replacement for nitroglycerine; trinitroamine oxide produces no toxic fumes upon detonation, and burns cleanly, making it well suitable for manufacturing explosives compositions for use in enclosed areas such as building, mines, tunnels, ect.

Safety Info: Use caution when handling nitric oxide, which rapidly reacts with air forming reddish fumes of nitrogen oxide, which are very toxic; use proper equipment when handling. Use caution when handling nitrogen dioxide gas which is very poisonous, use proper ventilation and avoid inhalation of the gas. Do not handle dry ice with bare hands, as it can cause frostbite. Use extreme care when handling high voltage equipment; always use non-conductive safety gloves when handling high voltage equipment, and wear eye protection and face mask.

Materials:

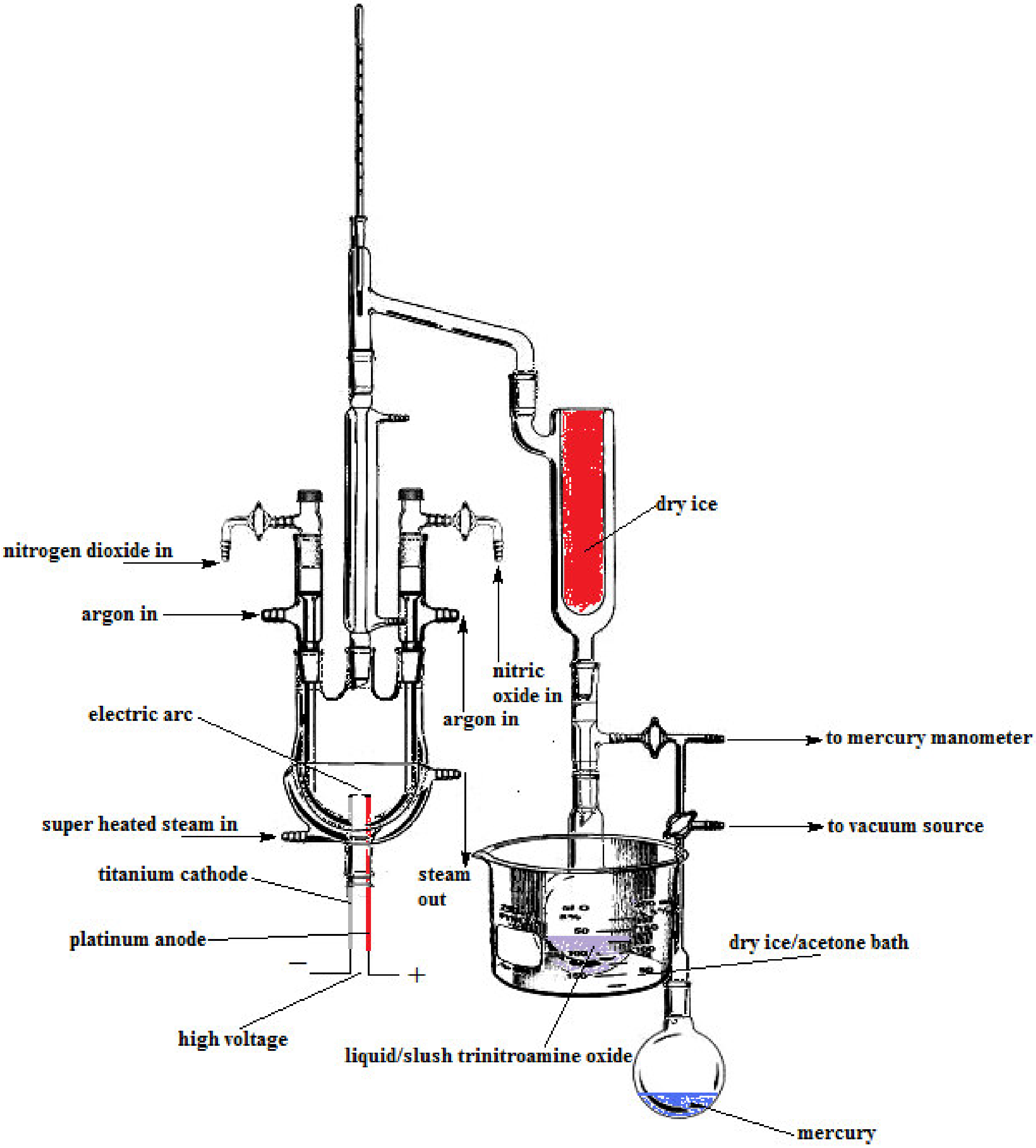
1. Dry argon gas from tank	3. 30 grams of dry nitric oxide
2. A fresh supply of dry ice chunks	4. 138 grams of dry nitrogen dioxide gas

Reaction Equation:



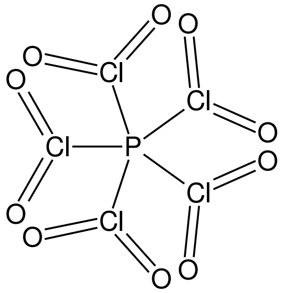
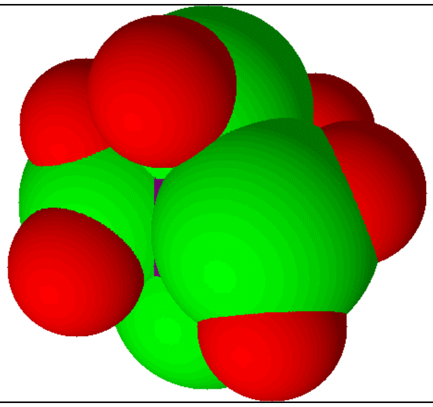
trinitroamine oxide

Preparation: Into an apparatus as illustrated below, flush the entire apparatus and purge the apparatus with dry argon gas to remove oxygen, and then turn on the vacuum and reduce the pressure inside the apparatus to 250mm of mercury. When the pressure inside the reaction apparatus reaches around 250mm of mercury, begin to pass super heated steam through the steam jacket around the reaction flask, to heat the inside of the reaction flask to around 150 Celsius. Turn on the power supply, to a voltage of 20,000 volts at 150 milliamps, and immediately begin to pass in dry nitric oxide gas and dry nitrogen dioxide gas at a flow rate of the nitric oxide of 15 mg per/minute, and 69 mg per/minute of the nitrogen dioxide gas until a total of 30 grams of nitric oxide, and 138 grams of nitrogen dioxide gas has been purged into the reaction flask. During the addition of the nitric oxide, and nitrogen dioxide gas, the electric arc and platinum anode will catalyze the addition of the nitric oxide to the nitrogen dioxide, whereby oxidation will take place forming the desired trinitroamine oxide, which under reduced pressure will vaporize and pass over into the dry ice condenser, where it will condense into a slushy liquid mass, from where it will fall into the receiver flask. After the proper amounts of nitric oxide and nitrogen dioxide have been added, immediately turn off the power supply, vacuum source, and turn of the argon gas purge. Then simply remove the receiver flask containing the slushy liquid mass of trinitroamine oxide and allow it to warm to room temperature in a sealed container. Once it has warmed to room temperature, store it in a sealed glass container in a refrigerator until use.



Setup for preparation of trinitroamine oxide.

Theoretical Preparation 15: pentachloryl-λ^5-phosphane

 O O O O Cl Cl Cl P Cl Cl O O O O O O pentachloryl-λ^5-phosphane	

Expanded structure	3D structure

Molecular data:

Molecular Formula = $Cl_5O_{10}P$
Formula Weight = 368.232762
Composition = Cl(48.14%) O(43.45%) P(8.41%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 365.767171 Da
Nominal Mass = 366 Da
Average Mass = 368.2328 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7890	Explodes when heated, or ignited.	Sensitive to shock, percussion, pressure, and friction; easily detonated. A primary explosive.	Chemically speaking, un-reactive. Keep out of contact with reducing agents.	Light lemon-yellowish red crystalline mass, or amorphous powder. Two allotropic forms exist, beta form: is a light yellowish red solid, melting point: Explodes; Alpha form, red amorphous solid, melting point: Explodes. Both forms are insoluble in water, and are slowly hydrolyzed by water. Both forms are insoluble in most organic solvents. Moderately solubility in liquid ammonia.

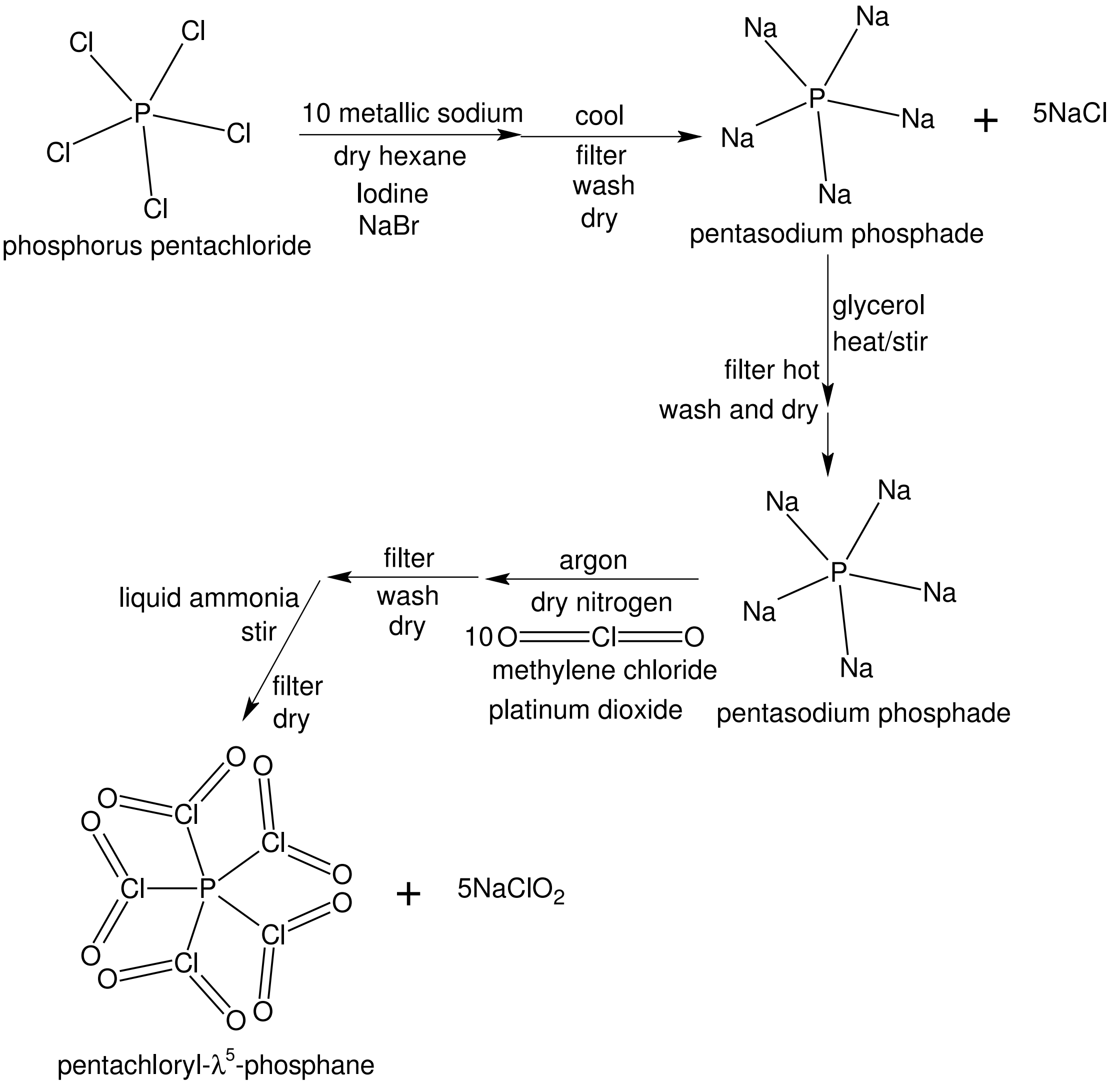
Estimated Compound summary: pentachloryl-λ^5-phosphane forms two allotropic forms, the beta and alpha forms. The allotropic forms prepared in this procedure is primarily the alpha form. The beta form is prepared by heating the alpha form in a vacuum to 150 Celsius for 24 hours. pentachloryl-λ^5-phosphane is a radical new inorganic explosive that resembles a mixture of phosphorus and a chlorate often seen in pyrotechnic mixtures, and are generally very dangerous to handle and deal with; however, pentachloryl-λ^5-phosphane has enough stability to warrant further investigation for use as a primary explosive in blasting caps and detonators mainly to replace the aging lead styphnate, and lead azide; as pentachloryl-λ^5-phosphane can be used in pure form without being diluted with inert fillers for stability. pentachloryl-λ^5-phosphane has remarkable explosive power, and yet is very easily detonated. It should however, never be mixed with powdered metals, or reducing agents, as these form very sensitive mixtures that can be detonated by simply hitting a small sample of the mixture with a wooden stick; however, when used in pure form, pentachloryl-λ^5-phosphane has remarkable stability, and can be stored without any real danger for years when kept pure and dry. pentachloryl-λ^5-phosphane can also be used in priming compositions for bullets and other shell casing primers.

Safety Info: Extinguish all flames and sources of ignition before using hexane, which is highly flammable. Use extreme care when handling metallic sodium, which can explode in contact with water. Wear gloves and proper eye protection when handling metallic sodium and avoid skin contact. Wear gloves when handling phosphorus pentachloride, and avoid contact with moisture and water. Use extreme caution when handling chlorine dioxide, and never use the gas pure as it may explode in the presence of heat and/or light; always dilute chlorine dioxide gas with an inert gas such as nitrogen, argon, ect., Use proper ventilation and avoid inhalation, ingestion, and skin contact when handling liquid ammonia.

Materials:

1. 2750 milliliters of dry hexane	7. 2700 milliliters of methylene chloride
2. 166 grams of metallic sodium	8. 1663 grams of chlorine dioxide
3. 12 grams of sodium bromide	9. 1 gram of platinum dioxide
4. 1 gram of pure iodine	10. Dry nitrogen gas from tank
5. 150 grams of phosphorus pentachloride	12. 3500 milliliters of dry liquid ammonia
6. 1200 milliliters of 99% glycerol	12. Dry argon from tank

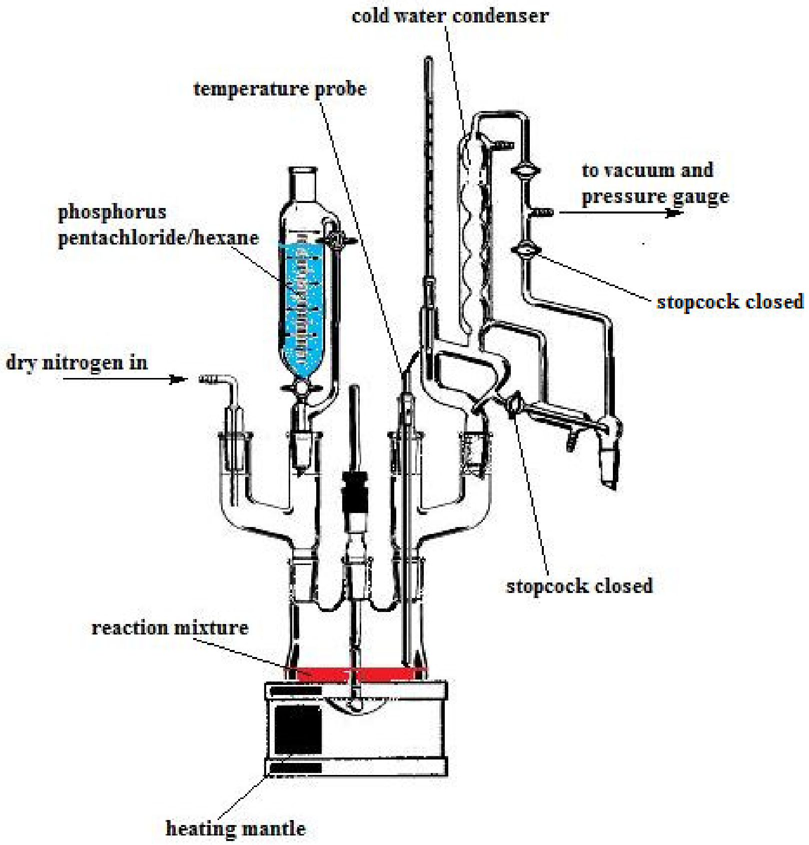
Reaction Equation:



Preparation:

Step 1: Preparation of pentasodium phosphade

Into a suitable three neck flask equipped with addition funnel, motorized stirrer, reflux condenser, and temperature probe, place 500 milliliters of dry hexane, and then add in, in small chunks or pieces, 166 grams of metallic sodium, followed by 12 grams of sodium bromide, and then followed by 1 gram of pure iodine. Thereafter, turn on the heating mantle, and carefully heat this mixture to 89 Celsius with rapid stirring. Note: purge the apparatus with dry nitrogen to remove oxygen and moisture, and then apply a vacuum of 500mm of mercury to reduce the pressure inside the 3-neck reaction flask. When the temperature inside the 3-neck flask reaches right around 89 Celsius, and a vacuum of 500mm of mercury, pour into the addition funnel a mixture of 150 grams of phosphorus pentachloride dissolved in 300 milliliters of dry hexane over a period of about 2 hours, while rapidly stirring the reaction mixture, and maintaining its temperature around 89 Celsius under a vacuum of 500mm of mercury. After all the phosphorus pentachloride has been added to the reaction mixture, continue to heat and reflux the reaction mixture at 89 Celsius under a moderate vacuum of 500mm of mercury for 6 hours with constant stirring. After 6 hours, remove the heat source, and the vacuum and allow the temperature of the reaction mixture to cool to room temperature, and remove the nitrogen purge and allow the pressure inside the reaction mixture to come to atmospheric pressure. Once it these conditions have been met, disassemble the apparatus, and then cap the 3-neck flask containing the reaction mixture and then store this 3-neck flask in a freezer for 1 hour. After 1 hour, filter-off all the insoluble material using suction filtration, and then wash this insoluble materials with three 150-milliliter portions of fresh hexane. After the washing, suction dry the filtered-off material as best as possible.

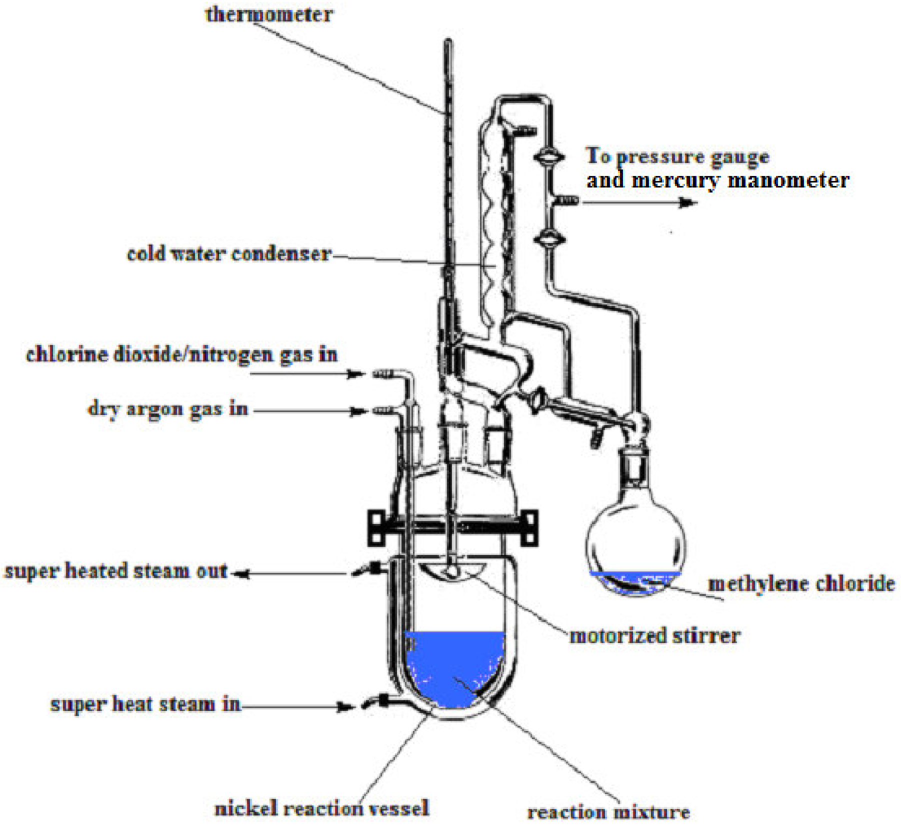


Setup for preparing pentasodium phosphide.

Now, into a large beaker, pour in 1200 milliliters of 99% glycerol, and then bring the temperature of this glycerol to 80 Celsius with stirring (use a hot plate/magnetic stirrer). When the temperature of the glycerol has reached 80 Celsius, slowly add in, in small portions at a time, the dried material obtained from the previous operation while maintaining the glycerol at 80 Celsius with rapid magnetic stirring. After all the dried material from the previous operation has been added, continue to heat the entire mixture for 4 hours at 80 Celsius with constant stirring. After 4 hours, quickly filter-off the insoluble mass, which will be the desired product of pentasodium phosphade using suction filtration, and then wash this filtered-off material with three 150-milliliter portions of dry hexane, and then suction dry. Thereafter, recover the dried desired product of pentasodium phosphade and store for step 2 (note: store in a desiccater over phosphorus pentoxide until use). Note: pentasodium phosphade may ignite spontaneously in moist air so use caution.

Step 2: Preparation of pentachloryl-λ^5-phosphane

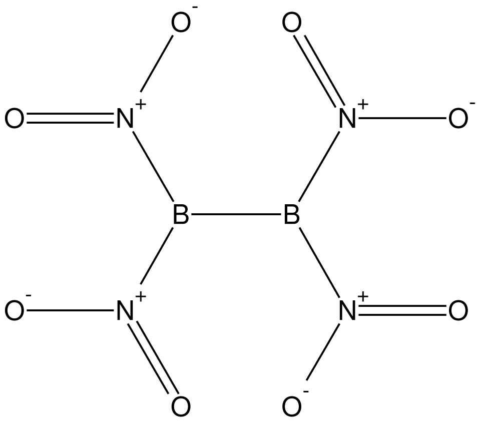
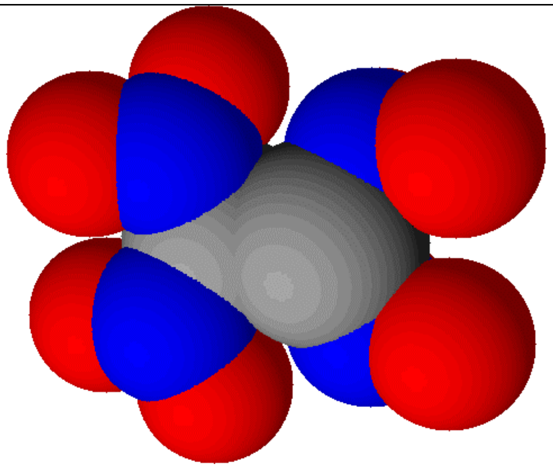
Into a suitable apparatus (utilizing a nickel reaction vessel plated with palladium) as illustrated below, place the dried product obtained from step 1, followed by 1200 milliliters of methylene chloride, and then followed by 1 gram of platinum dioxide. Thereafter, begin an argon purge to flush out all the oxygen in the apparatus, and to obtain a pressure of 500 ATM's. Thereafter, begin to pass super heated steam into the jacked surrounding the reaction vessel, and bring the temperature inside the reaction vessel to 180 Celsius. When the temperature reaches 180 Celsius begin to flush in a gas mixture of nitrogen/and chlorine dioxide of a ratio of 5 parts nitrogen gas to 1 part chlorine dioxide gas at a flow rate of 50 mg per/minute, and continue to flush in this nitrogen/chlorine dioxide gas mixture until 1663 grams of chlorine dioxide has been added. Once all the chlorine dioxide has been added, turn off the nitrogen gas source, and then continue to heat the reaction mixture to 180 Celsius while continuing to purge the apparatus with dry argon gas under a pressure of 500 ATM's for 3 hours with constant stirring of the motorized stirrer to act as a fan to circulate the gasses and vapors. After 3 hours, remove the steam source, and release the pressure gauge to bring the pressure in the reaction vessel back to atmospheric pressure, and then allow the contents in the reaction vessel to cool to room temperature. Note: continue to purge the apparatus with dry argon gas during the cool down period.



Setup for addition of chlorine dioxide and nitrogen.

Now, once the contents in the reaction flask have cooled to room temperature, disassemble the apparatus, and then quickly suction filter the reaction mixture to recover the insoluble products of sodium chlorite and the desired product of pentachloryl-λ^5-phosphane. Once recovered in a Buchner funnel, wash the entire filtered mass with three 500-milliliter portions of dry hexane, and then suction dry as best as possible. Immediately thereafter, place the suction dried mass in the Buchner funnel into a large beaker filled with 3500 milliliters of dry liquid ammonia, and then rapidly blend the mixture to dissolve the desired product for about 1 hour. After 1 hour, quickly suction filter the liquid ammonia mixture to recover the insoluble sodium chlorite, and then place the liquid ammonia containing the dissolved desired product of pentachloryl-λ^5-phosphane, and then allow the liquid ammonia to evaporate until dry solid remains. Note: use maximum ventilation. Thereafter, place this left over dry solid product into a Buchner funnel, and then wash the desired product with three 500-milliliter portions of dry methylene chloride using suction, and then suction dry the desired product of pentachloryl-λ^5-phosphane as best as possible, and then place this suction dried product into an oven and bake at 40 Celsius for 6 hours. After 6 hours, turn off the oven, and allow the desired product to cool to room temperature. Finely, remove the desired product of pentachloryl-λ^5-phosphane, and store in a dessicator over phosphorus pentoxide until use.

Theoretical Preparation 16: Tetranitrodiborane; TNDB; Nitro Boron

 tetranitrodiborane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $B_2N_4O_8$

Formula Weight = 205.644

Composition = B(10.51%) N(27.24%) O(62.24%)

Molar Refractivity = 32.99 ± 0.3 cm^3

Molar Volume = 123.5 ± 3.0 cm^3

Parachor = 340.2 ± 4.0 cm^3

Index of Refraction = 1.446 ± 0.02

Surface Tension = 57.4 ± 3.0 dyne/cm

Density = 1.664 ± 0.06 g/cm^3

Dielectric Constant = Not available

Polarizability = 13.08 ± 0.5 $10^{-24}cm^3$

Monoisotopic Mass = 205.990225 Da

Nominal Mass = 206 Da

Average Mass = 205.644 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6189; estimated using NMR spectrum	Non-flammable; will burn violently if mixed with reducing agents and ignited.	Non-sensitive to shock, percussion, heat, sparks, flame, and friction. Very stable. Requires blasting cap or detonator for initiation. A secondary explosive.	Generally non-reactive; slowly hydrolyzed by water; completely hydrolyzed by boiling water. Can be reduced using traditionally means.	Colorless, odorless crystalline mass or powder. Melting point: 56 Celsius, Boiling point: 189 Celsius under reduced pressure. Impure grades may have a slight yellowish tint. Insoluble in water

				and most organic solvents.

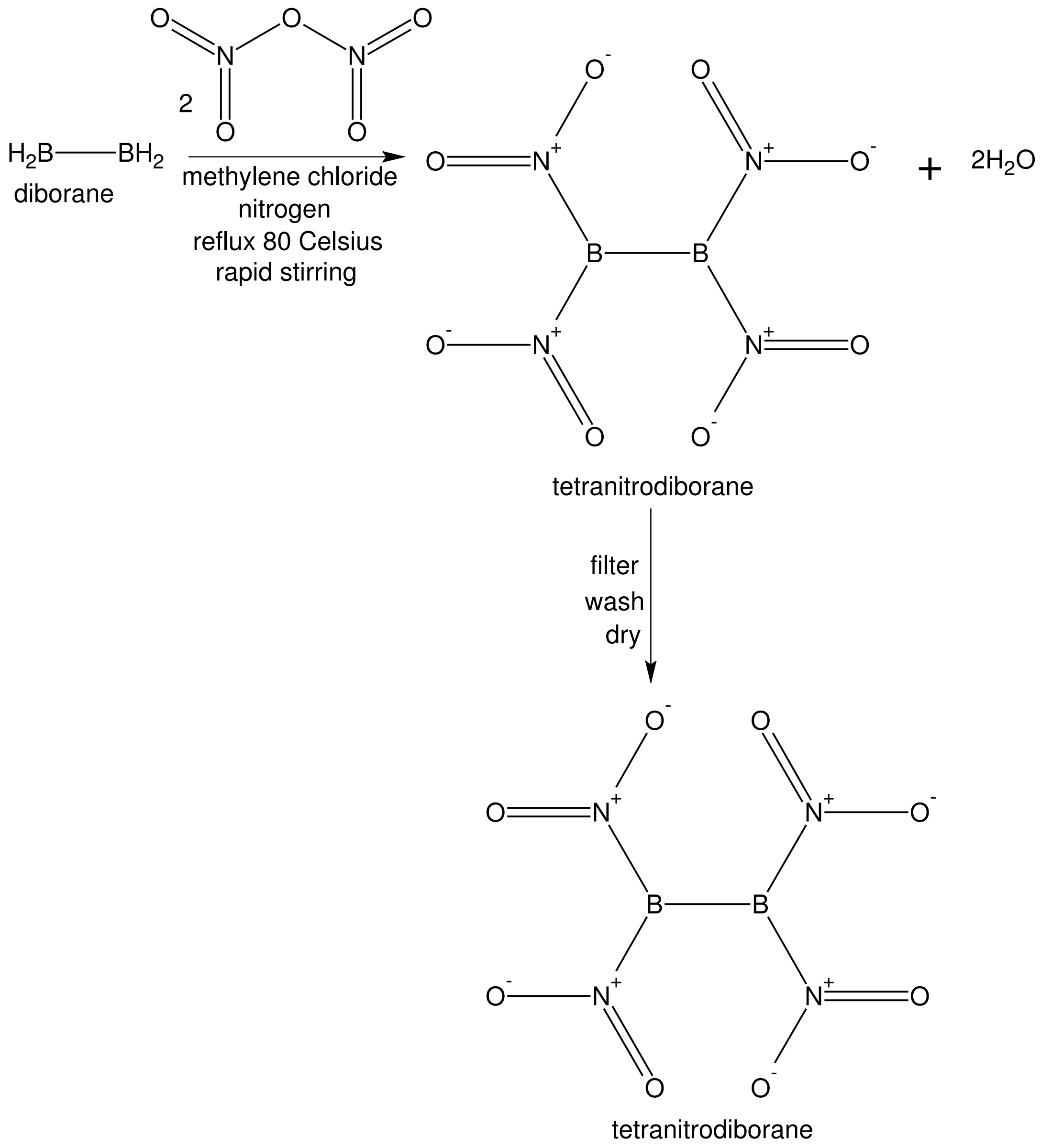
Estimated Compound summary: Tetranitrodiborane is an interesting boron containing high explosive with remarkable stability and properties, its low melting point allows it to be mixed with other explosives, fillers, and compounds to form explosive compositions for use in manufacturing shape charges for a variety of commercial and military operations; manufacturing explosive shells for mortars and artillery, and for making warheads for rockets. Because of its low melting point, it does not show any potential for use in explosive munitions such as high velocity projectiles, or space based munitions or space operations. Tetranitroborane shows interesting effects for use in fireworks, as it burns with great brilliance when admixed with powdered metals producing a wide variety of color effects that explode with brilliance when ignited.

Safety Info: Use care when handling dinitrogen pentoxide, which reacts with water forming nitric acid; the reaction can be violent so use care. Use caution when handling diborane gas, which is spontaneously combustible in air. Always dilute it with an inert gas when using. Extinguish all flames and sources of ignition when handling hexane, which is highly volatile and flammable.

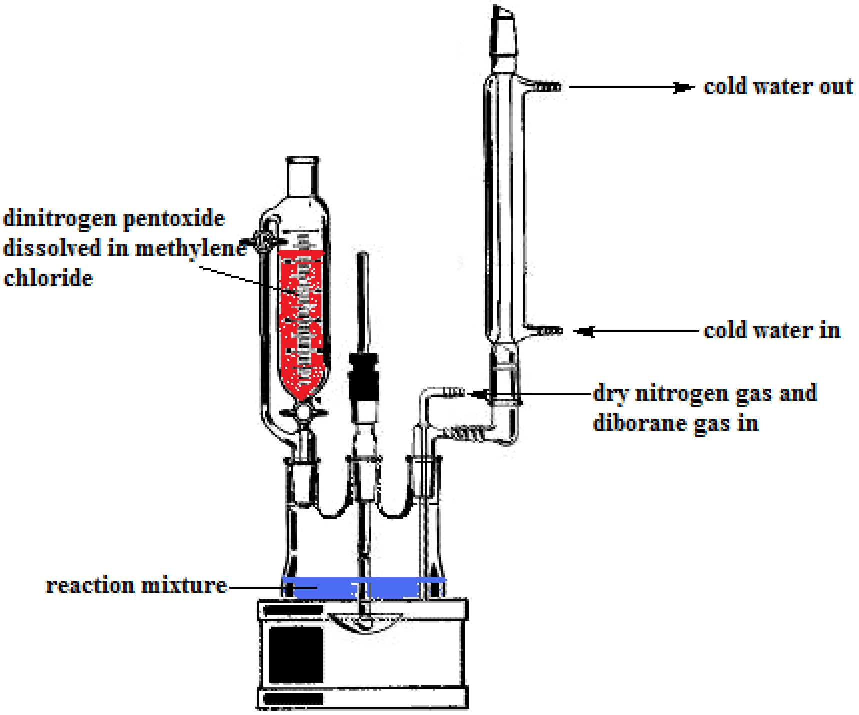
Materials:

1. 1750 milliliters of methylene chloride	4. 450 milliliters of dry hexane
2. 388 grams of dinitrogen pentoxide (prepared by dehydrating concentrated nitric acid with phosphorus pentoxide in the methylene chloride)	5. Nitrogen gas from tank
3. 46 grams of diborane gas	

Reaction Equation:

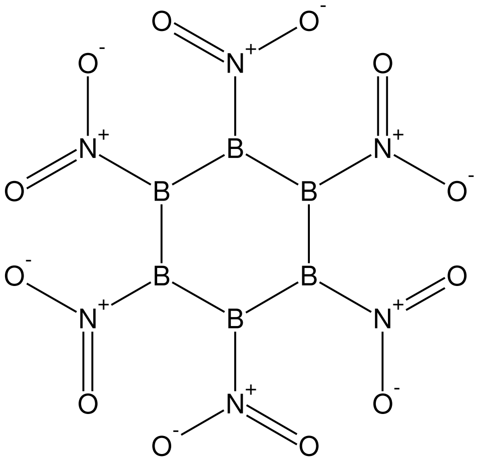
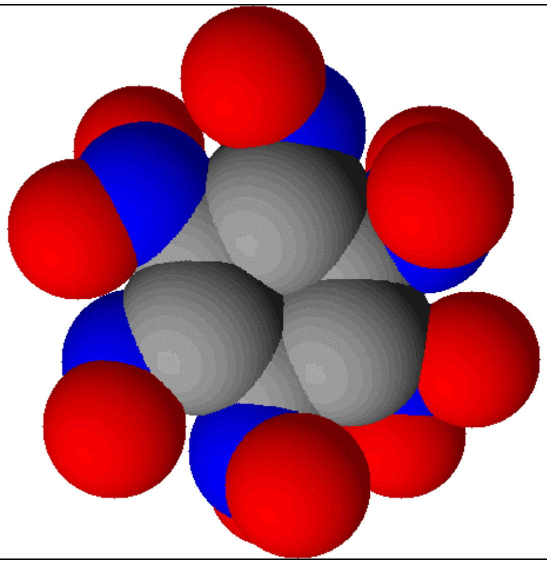


Preparation: Into an apparatus as illustrated below, pour in 750 milliliters of methylene chloride, and then pour into the addition funnel, ½ half of a mixture prepared by dissolving 388 grams of dinitrogen pentoxide into 1000 milliliters of methylene chloride. Thereafter, begin stirring the methylene chloride in the 3-neck flask and turn on the heating mantle and heat this methylene chloride to 80 Celsius. When the temperature reaches 80 Celsius begin a fast drip of the dinitrogen pentoxide/methylene chloride mixture in the addition funnel into the 3-neck flask, while rapidly stirring. Immediately thereafter begin to bubble in diborane/nitrogen gas at a flow rate of 150 mg per/minute until 46 grams of diborane gas has been bubbled into the reaction mixture. Now, before half the diborane gas has been added, pour the remaining ½ half of the dinitrogen pentoxide/methylene chloride mixture into the addition funnel and continue a fast drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while rapidly stirring the reaction mixture and maintaining its temperature at 80 Celsius under reflux. Note: all of the dinitrogen pentoxide/methylene chloride mixture in the addition funnel will be added to the reaction mixture before all the diborane gas has been added. After all the dinitrogen pentoxide/methylene chloride mixture in the addition funnel has been added, continue to rapidly stir the reaction mixture while maintaining a reflux at 80 Celsius for 4 hours. Note: before this 4 hour time period has been reached, all of the desired diborane would have been added, but continue to bubble into the reaction mixture dry nitrogen gas from tank. After 4 hours, remove the nitrogen gas purge, and remove the heat source, and allow the reaction mixture to cool to room temperature, but turn off the stirrer during the cool down period. Note: the reaction mixture will be composed of two layers, an upper layer will be the water by-product, and the bottom phase will contain the methylene chloride, and bulk of the insoluble desired product. When the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter-off the insoluble desired product of tetranitrodiborane using suction filtration, and then wash the filtered-off product with three 150-milliliter portions of dry hexane, and then suction dry thoroughly for 1 hour. Thereafter, store the dried product in a dessicator over concentrated sulfuric acid.


cold water out
dinitrogen pentoxide dissolved in methylene chloride
cold water in
dry nitrogen gas and diborane gas in
reaction mixture

Setup for nitration of diborane.

Theoretical Preparation 17: 1,2,3,4,5,6-hexanitrocyclohexaborane; KNCHB; 6-Nitrocycloborane

 1,2,3,4,5,6,hexanitrocyclohexaborane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $B_6N_6O_{12}$
Formula Weight = 340.899
Composition = B(19.03%) N(24.65%) O(56.32%)

Molar Refractivity = 57.06 ± 0.4 cm^3
Molar Volume = 215.7 ± 5.0 cm^3
Parachor = 558.0 ± 6.0 cm^3
Index of Refraction = 1.441 ± 0.03
Surface Tension = 44.7 ± 5.0 dyne/cm
Density = 1.57 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 22.62 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 342.013255 Da
Nominal Mass = 342 Da
Average Mass = 340.899 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7890	Non-flammable, but burns violently when mixed with powdered metals and reducing agents, and ignited.	Non-sensitive to shock, percussion, friction, pressure, and heat. Requires blasting cap or detonator for initiation. A secondary explosive.	Has amazing stability and inertness towards many compounds; however, will decompose when added to boiling water.	Colorless, odorless crystals or white powder or granules. Melting point: 127 Celsius; Boiling point Decomposes above 350 Celsius. Relatively insoluble in water, and slowly hydrolyzed by it, insoluble in most organic solvents.

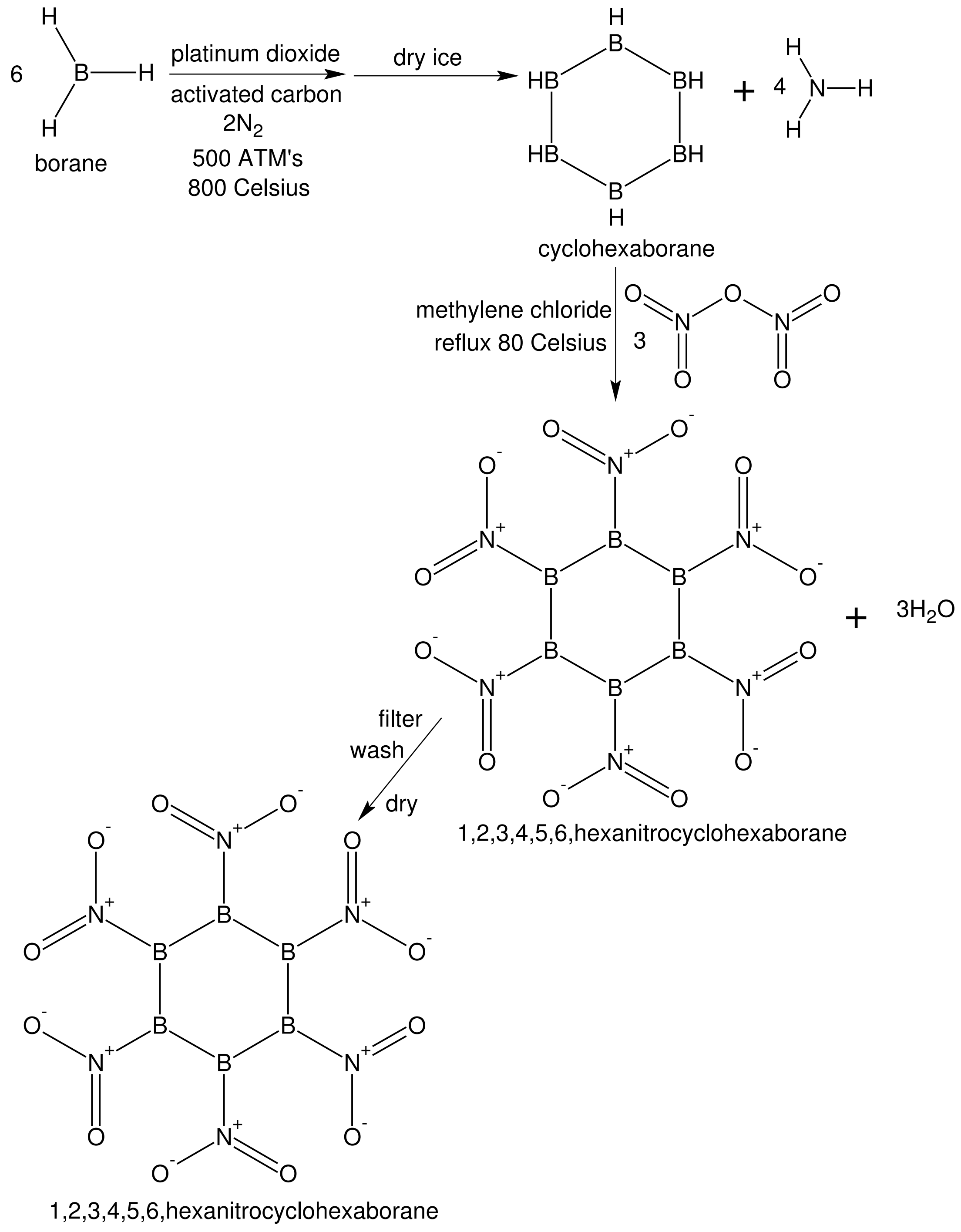
Estimated Compound summary: 1,2,3,4,5,6-hexanitrocyclohexaborane is an amazing and interesting cyclic boron compound that possesses great explosive power. The compound is useful in making a variety of explosive munitions such as mortar rounds, artillery shells, high velocity tank fired projectiles, rocket and missile warheads, and in manufacturing shape charges. It can be alloyed with other high explosives such as TNT, RDX, or PETN in making a variety of explosive compositions for commercial and military use. 1,2,3,4,5,6-hexanitrocyclohexaborane can be used in making powerful shape charges by simply melting it down, and then casting it into molds. For long-term use, 1,2,3,4,5,6-hexanitrocyclohexaborane should be alloyed with other high explosives such as TNT or RDX for use in making land mines, and underwater explosive munitions such as torpedoes to protect 1,2,3,4,5,6-hexanitrocyclohexaborane due to its moisture sensitivity; however 1,2,3,4,5,6-hexanitrocyclohexaborane has a half life of 45 days when exposed to water at room temperature. Hence for long term storage in munitions that may be exposed to moisture it needs to be stabilized with water resistant explosives like TNT, or RDX. 1,2,3,4,5,6-hexanitrocyclohexaborane can be used in making a variety of pyrotechnics, and for making fireworks, as it burns with great brilliance when admixed with powdered metals and ignited. When alloyed with powdered aluminum and a plasticizer, it makes a powerful high performance rocket fuel for a variety of commercial and military use.

Safety Info: Use extreme care when handling borane gas, which is spontaneously combustible in air. Extinguish all flames and sources of ignition before handling hexane, which is highly volatile and flammable. Use caution and wear gloves and eye protection when handling dinitrogen pentoxide.

Materials:

1. 50 grams of platinum dioxide	5. 1700 milliliters of methylene chloride
2. 150 grams of activated carbon	6. 449 grams of dinitrogen pentoxide
3. 106 grams of dry borane gas	7. 450 milliliters of dry hexane
4. Dry nitrogen from tank	

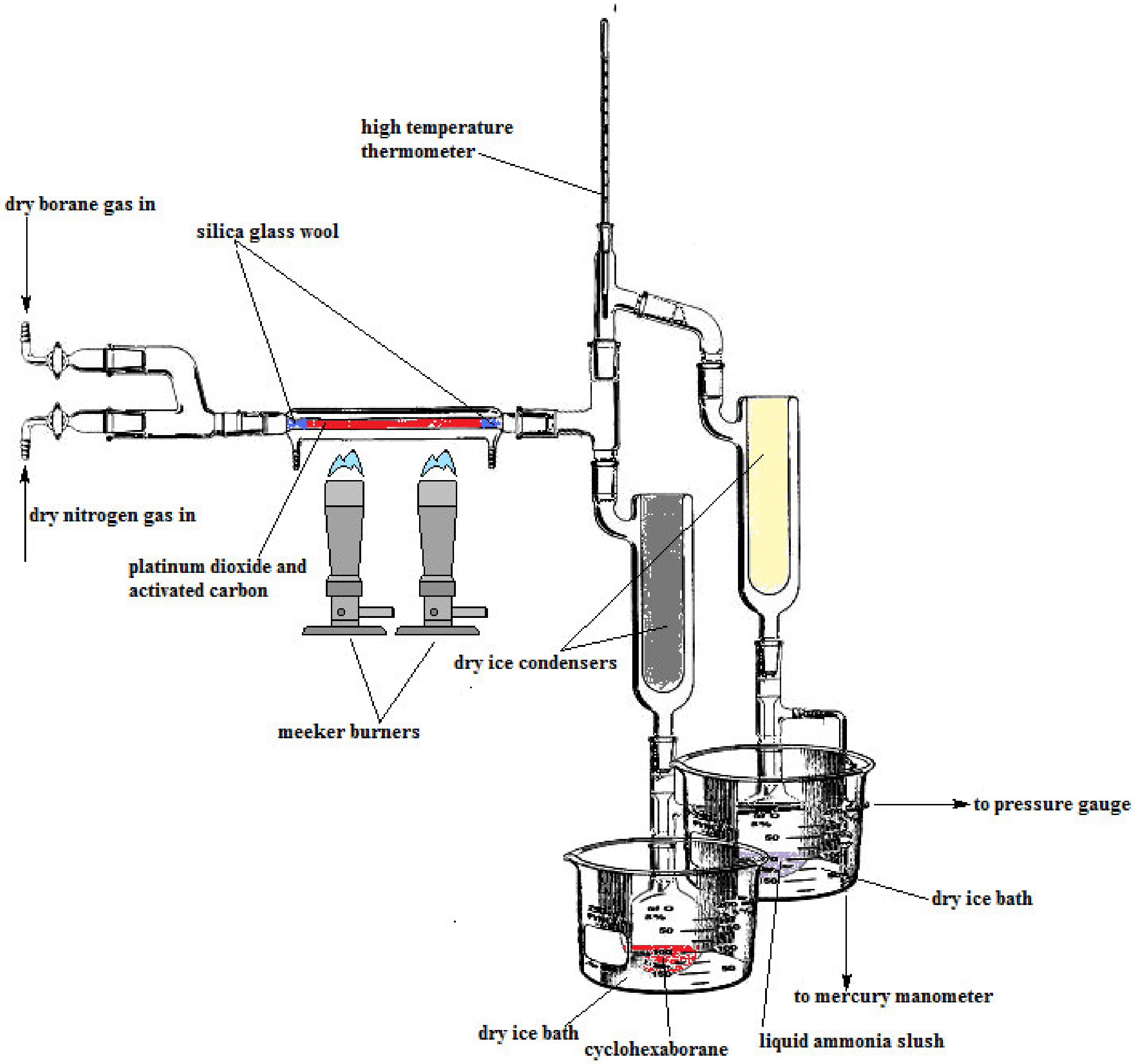
Reaction Equation:



Preparation:

Step 1: Preparation of cyclohexaborane

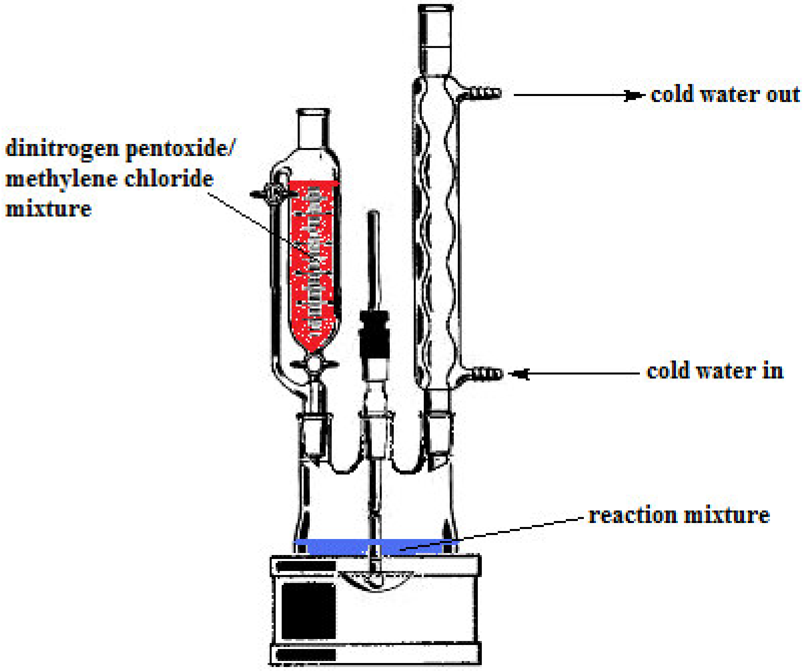
Into an apparatus as illustrated below, mix dry, 50 grams of platinum dioxide with 150 grams of activated carbon, and then place into the quartz/silica condenser tube, and then place silica glass wool plugs at each end to hold in place the catalyst mixture. Thereafter, begin a dry nitrogen purge and bring the pressure in the apparatus to 500 ATM"s. Thereafter, turn on the meeker burners and bring the temperature of the glass condenser tube to 800 Celsius. When the temperature in the glass condenser tube reaches 800 Celsius and the pressure inside the apparatus reaches 500 ATM's, begin to pump into the apparatus, dry borane gas at a flow rate of 100 mg per/minute. Continue to pump in dry borane gas until 106 grams of dry borane gas has been pumped in, along with 71.6 grams of dry nitrogen gas. After all the dry borane gas has been pumped in, continue to purge the apparatus with dry nitrogen gas and then remove the pressure release system, and bring the pressure inside the apparatus back to atmospheric pressure, and reduce the heat to 400 Celsius, and hold at this point for 1 hour. After 1 hour, remove the heat source, and allow the glass condenser tube's temperature to cool to room temperature while continuing to purge the apparatus with dry nitrogen gas. When room temperature is achieved, allow the apparatus to stand for 6 hours, while continuing the purge the apparatus with dry nitrogen gas from tank. After 6 hours, turn off the nitrogen purge, and then disassemble the apparatus, and place a glass stopper on the flask containing the cyclohexaborane, and store in a freezer until use.



Setup for the polymerization of borane. Note: the left portion of the apparatus is made of high temperature resistant quartz/silica glassware.

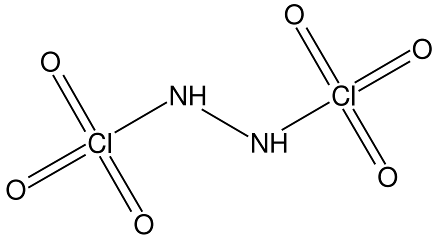
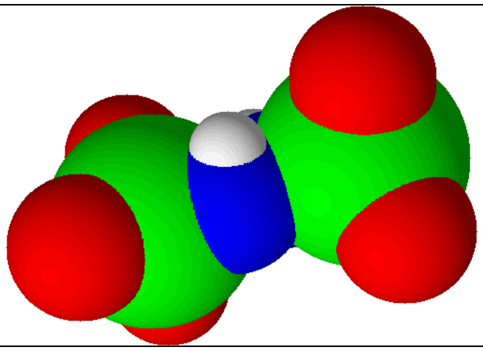
Step 2: Preparation of 1,2,3,4,5,6-hexanitrocyclohexaborane

Into an apparatus as illustrated below, pour in 500 milliliters of methylene chloride, and then add in the cyclohexaborane obtained from step 1, and then pour into the addition funnel, 1/3 of a mixture prepared by dissolving 449 grams of dinitrogen pentoxide into 1200 milliliters of methylene chloride. Thereafter, begin stirring the methylene chloride in the 3-neck flask and turn on the heating mantle and heat this methylene chloride to 80 Celsius. When the temperature reaches 80 Celsius begin a fast drip of the dinitrogen pentoxide/methylene chloride mixture in the addition funnel into the 3-neck flask, while rapidly stirring. After this 1/3 of the dinitrogen pentoxide/methylene chloride mixture has been added, pour into the addition funnel another 1/3 of the dinitrogen pentoxide/methylene chloride mixture and continue a fast drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while rapidly stirring the reaction mixture and maintaining its temperature at 80 Celsius under reflux. After this 1/3 of the dinitrogen pentoxide/methylene chloride mixture has been added, pour in the remaining dinitrogen pentoxide/methylene chloride mixture into the addition funnel and continue a rapid drip, while rapidly stirring the reaction mixture and maintaining its temperature at 80 Celsius under reflux. After all the dinitrogen pentoxide/methylene chloride mixture in the addition funnel has been added, continue to rapidly stir the reaction mixture while maintaining a reflux at 80 Celsius for 4 hours. After 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature, but continue to stir the reaction mixture rapidly during this cool down period. When the temperature of the reaction mixture has cooled to room temperature, turn off the stirrer, and allow the reaction mixture to stand at room temperature for 1 hour. After 1 hour, the reaction mixture will be composed of two layers, an upper water layer, and a lower methylene chloride layer containing the bulk of the desired product. After 1 hour, disassemble the apparatus, and filter the reaction mixture using suction filtration to recover the insoluble desired product of 1,2,3,4,5,6-hexanitrocyclohexaborane, and then wash the filtered-off product with three 150-milliliter portions of dry hexane, and then suction dry thoroughly for 1 hour. Thereafter, store the dried product in a dessicator over concentrated sulfuric acid until use.



Setup for nitration of cyclohexaborane.

Theoretical Preparation 18: *N*'-perchlorylperchloric hydrazide; N’PCPH, Perchloryl hydrazine

 *N*'-perchlorylperchloric hydrazide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_2Cl_2N_2O_6$

Formula Weight = 196.93168

Composition = H(1.02%) Cl(36.01%) N(14.22%) O(48.75%)

Molar Refractivity = Not available

Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 195.928991 Da
Nominal Mass = 196 Da
Average Mass = 196.9317 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6570; estimated using infrared spectrum	Non-flammable; ignites violently when mixed with reducing agents and ignited. Mixtures with powdered aluminum explode violently when ignited.	Non-sensitive. Stable to shock, percussion, heat, and friction. Requires blasting cap or detonator for initiation. A secondary explosive.	Non-reactive. Very chemically inert. Does not react with moisture or water even boiling water.	Light reddish crystalline powder, granules, or masses. Melting point: 103 Celsius, Boiling point: 309 Celsius. Decomposes when heated above 350 Celsius. Insoluble in water, and most organic solvents. Limited solubility in liquid ammonia.

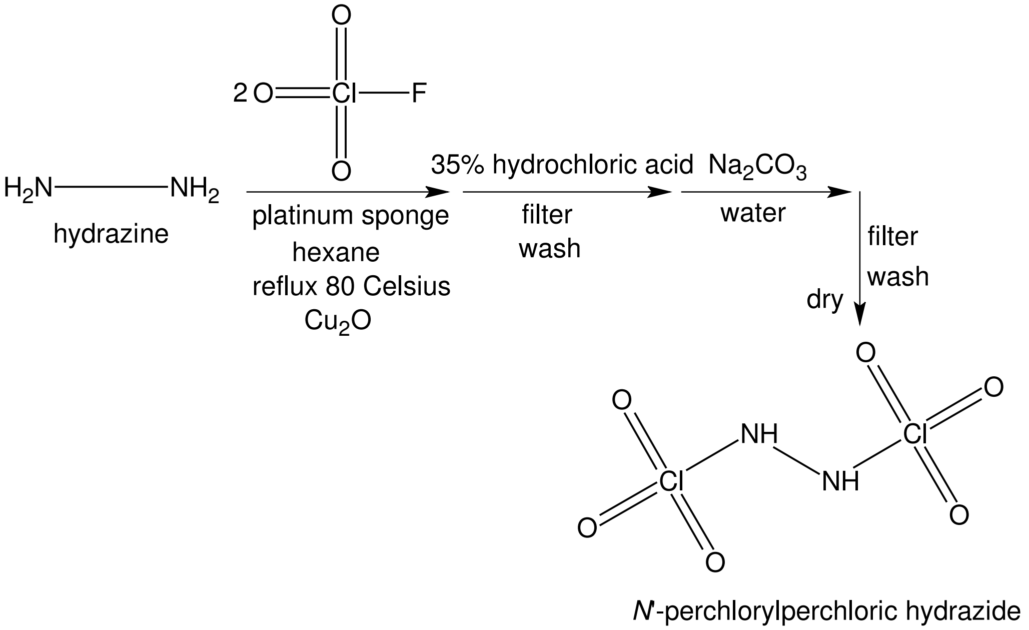
Estimated Compound summary: *N'*-perchlorylperchloric hydrazide is an interesting hydrazine based perchloryl explosive with tremendous stability and properties. It has remarkable stability and is heat resistant up to 350 Celsius, and is virtually chemically inert to many compounds; however, the hydrazine portion is capable of reducing transition metal oxides from their higher states down to their lower states. It has a relatively low melting point making it suitable to be melted down and poured into molds for making shape charges, fillers for mortar and artillery shells, and when melted down with other explosives for making powerful missile warheads. The explosive is multiple purpose and can be used in making blasting agents for mining soft or hard rock, for making bombs, and other explosive munitions for many commercial and military operations. *N'*-perchlorylperchloric hydrazide shows tremendous properties for use as a rocket fuel when mixed with reducing agents (other then aluminum) and cured with epoxy. Even though its detonating velocity is lower then most high explosives, its thrust to weight ratio is enormous and can be used to make powerful high performance rocket fuels for anti-aircraft missiles. *N'*-perchlorylperchloric hydrazide shows remarkable use for making pyrotechnic mixtures for various commercial and military operations, as it burns with great brilliance when admixed with reducing agents and ignited. The explosive has a tendency to explode violently when mixed with powdered aluminum and ignited by spark or flame. *N'*-perchlorylperchloric hydrazide forms violently exploding devices when mixed with powdered metals such as magnesium or iron, so it can be used in making fireworks such as firecrackers, or it can be used as a bursting charge for aerial display fireworks. The explosive burns with excellent brilliance of color, light and sound when mixed with reducing agents and cured with epoxy for making beautiful fireworks such as fountains and cones. It can also be used as a replacement for black powder in lifting charges for propelling aerial firework shells, as it has great heaving power when deflagrated, and has 3 times the lifting power of conventional black powder.

Safety Info: Extinguish all flames when handling hexane, which is highly volatile and flammable. Use care when handling anhydrous hydrazine and never heat it in the presence of oxygen as explosion may result. Wear gloves and use proper ventilation when handling concentrated hydrochloric acid, which is very volatile and emits suffocating fumes. Use extreme caution when handling perchloryl fluoride, which is very refractive and has a tendency to explode in contact with certain organic material.

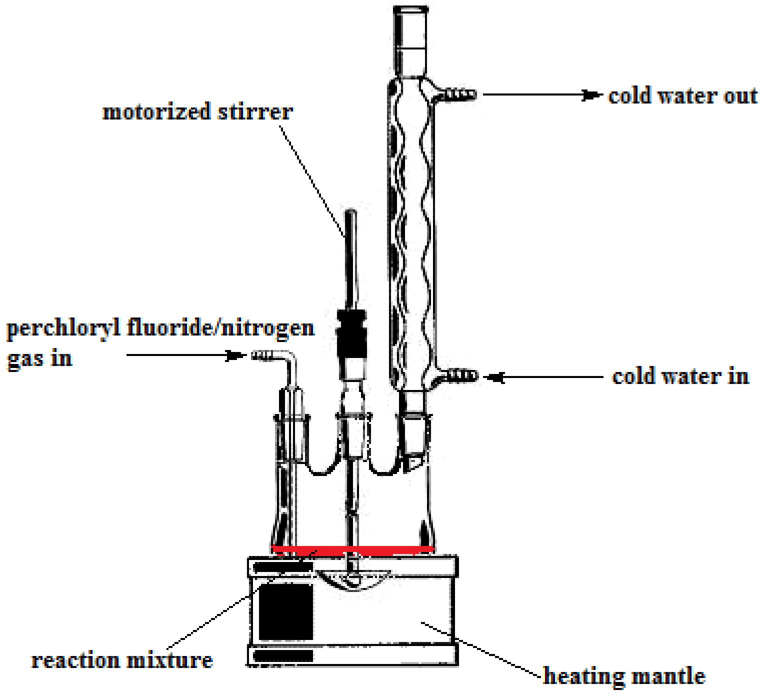
Materials:

1. 600 milliliters of dry hexane	6. 50 milliliters of concentrated hydrochloric acid
2. 106 grams of anhydrous hydrazine	7. 1500 milliliters of a 15% sodium carbonate solution
3. 5 grams of platinum sponge	8. 450 milliliters of methylene chloride
4. 12 grams of copper-I-oxide	9. 678 grams of perchloryl fluoride gas
5. Dry nitrogen gas from tank	10. 50 milliliters of concentrated hydrochloric acid

Reaction Equation:

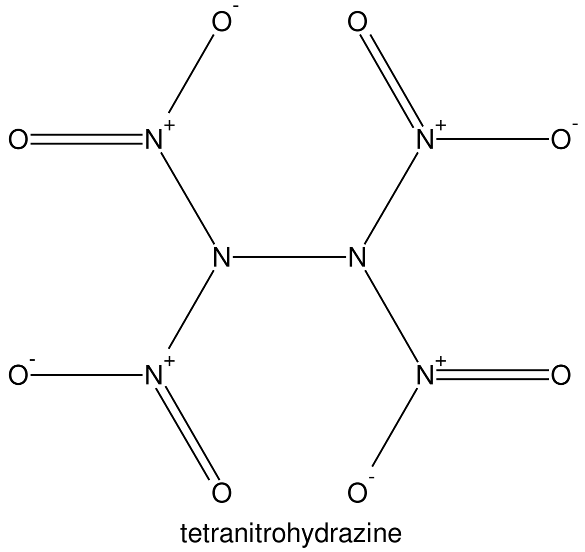
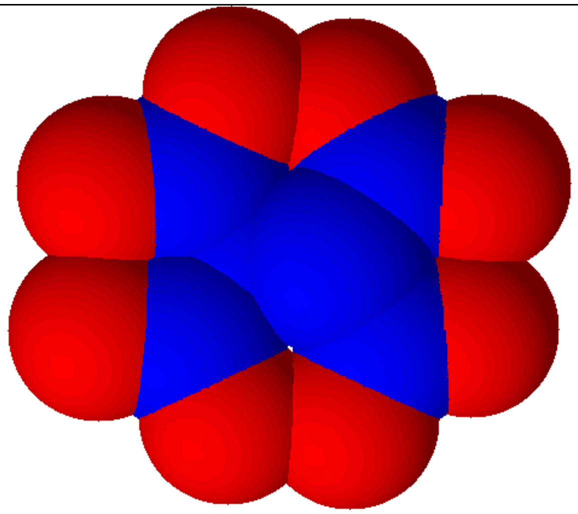


Preparation: Into a suitable apparatus as illustrated below, purge the apparatus with dry nitrogen gas to flush out moisture and oxygen, and then pour into the 3-neck flask 600 milliliters of dry hexane, followed by 106 grams of anhydrous hydrazine, followed by 5 grams of platinum sponge, and then followed by 12 grams of copper-I-oxide. Thereafter heat the contents in the 3-neck flask to 80 Celsius and begin a gentle reflux with rapid stirring. When the temperature inside the 3-neck flask reaches 80 Celsius, begin to bubble into the reaction mixture dry perchloryl fluoride gas at a flow rate of 50 mg per/minute and a nitrogen purge of 500 mg per/minute. Continue to bubble in dry perchloryl fluoride gas until 678 grams of perchloryl fluoride has been added. During the addition of the perchloryl fluoride gas, rapidly stir the reaction mixture and maintain a gentle reflux at 80 Celsius while continuing to purge the apparatus with nitrogen gas. Once all the perchloryl fluoride gas has been added, continue to stir the reaction mixture rapidly while maintaining a gentle reflux of 80 Celsius for 4 hours, but continue the nitrogen gas purge. After 4 hours, remove the nitrogen purge, remove the heat source, and allow the reaction mixture to cool to room temperature. When the reaction mixture has cooled to room temperature stop the stirring, and allow the reaction mixture to stand in a freezer for 6 hours. Note: simply use three glass stoppers to seal the 3-neck flask containing the reaction mixture. After 6 hours, remove the sealed three-neck flask containing the reaction mixture, and then add in 50 milliliters of concentrated hydrochloric acid to dissolve out the copper-I-oxide. Once the copper-I-oxide has dissolved in the acid, filter the reaction mixture using suction filtration to recover the insoluble intermediate product, and then pluck out the platinum sponge and save for another run or different procedure. Thereafter, wash this filtered-off intermediate with three 500-milliliter portions of hot water, and then suction dry as best as possible. Now, pour the filter cake into a large beaker and then add in 1500 milliliters of a 15% sodium carbonate solution, and then stir the entire mixture for 2 hours at room temperature. After 2 hours, filter off the insoluble product using suction filtration, and then wash with three 150-milliliter portions of methylene chloride, and then suction dry thoroughly. Thereafter, place the dried filtered-off product into an oven and bake at 90 Celsius for 3 hours. After 3 hours, remove the desired product of *N'*-perchlorylperchloric hydrazide from the oven and allow it to cool to room temperature.



Setup for addition of perchloryl fluoride to hydrazine.

Theoretical Preparation 19: tetranitrohydrazine; TNH-X

	
Expanded structure	3D structure

Molecular data:
Molecular Formula = N_6O_8
Formula Weight = 212.0354
Composition = N(39.63%) O(60.37%)
Molar Refractivity = 33.58 ± 0.3 cm^3
Molar Volume = 94.1 ± 3.0 cm^3
Parachor = 317.7 ± 4.0 cm^3
Index of Refraction = 1.631 ± 0.02
Surface Tension = 129.6 ± 3.0 dyne/cm
Density = 2.251 ± 0.06 g/cm^3

Dielectric Constant = Not available
Polarizability = 13.31 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 211.977761 Da
Nominal Mass = 212 Da
Average Mass = 212.0354 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
8170	Non-flammable; will not ignite violently even when mixed with reducing agents.	Non-sensitive to shock, percussion, heat, or friction. Requires blasting cap or detonator for initiation. A secondary explosive	Non-reactive. Can be reduced from finely divided tin and hydrochloric acid.	Colorless, syrupy liquid, Melting point: -45 Celsius; Boiling point: 190 Celsius under reduced pressure. Explodes when rapidly heated to 450 Celsius. Insoluble in water, but moderately soluble in methylene chloride, hexane, and 99% methanol, but relatively insoluble in ether

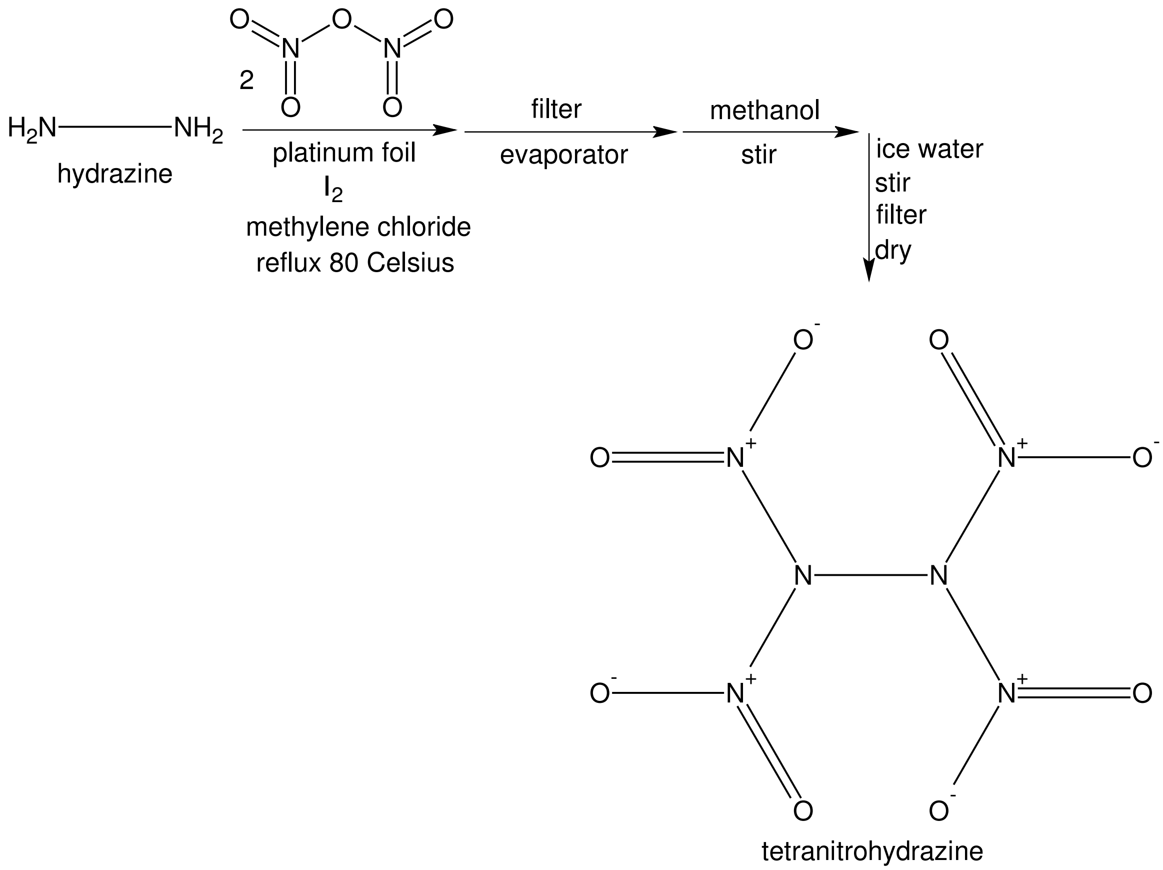
Estimated Compound summary: tetranitrohydrazine is an amazing syrupy liquid explosive, with tremendous power. It is one of the only known liquid explosives that is very resistant to shock, percussion, heat, and friction. It can be stored for years under moderate conditions without fear of premature ignition or detonation. Tetranitrohydrazine is designed to completely replace the old aging nitroglycerine in explosives for a variety of commercial and military operations, mainly in the field of demolitions. It can be used either straight as a liquid, where it is poured into wax covered cardboard tubes, or it can be absorbed by diatomaceous earth, finely divided silicon dioxide, powdered aluminum, or aluminum oxide to form explosive chunks that are then dead pressed into any desired vessel including mortar rounds, artillery shells, tubes for blasting rock, or any desired bomb casing. tetranitrohydrazine, can also be used in place of nitroglycerine for making double based high performance gun propellants. Also, unlike nitroglycerine and similar liquid explosives of the polyalcohol type, tetranitrohydrazine is relatively non-toxic, and does not produce headaches or migraines; it's medicinal value is unknown, but it may have use as a vasodilator.

Safety Info: Use caution when handling iodine, which is volatile and emits corrosive purple fumes; avoid inhalation and ingestion. Use extreme care when handling dinitrogen pentoxide, which is very reactive and reacts vigorously with water forming nitric acid. Use extreme caution when handling anhydrous hydrazine, and never boil or distill it in the presence of air as explosion may result. 99% methanol is toxic so avoid skin contact, ingestion, and inhalation.

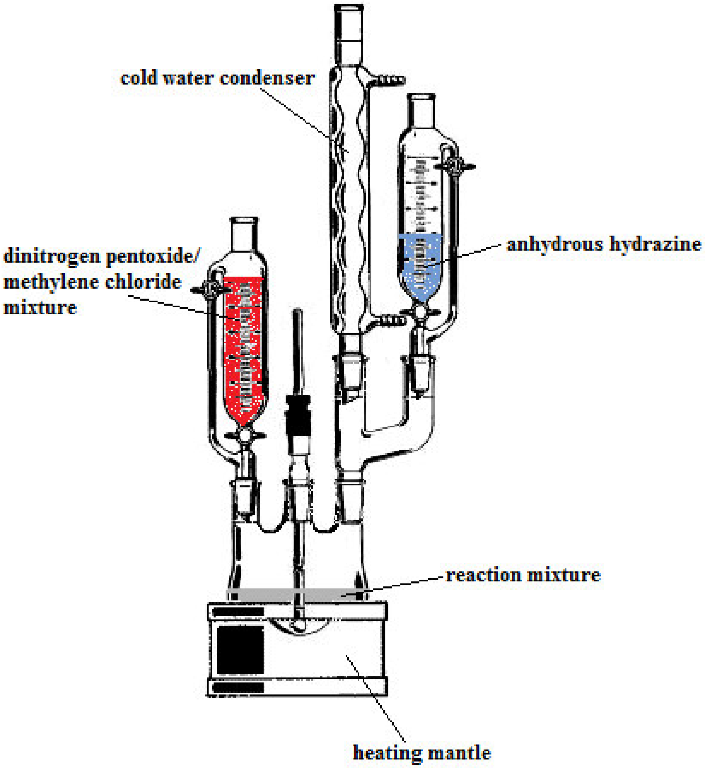
Materials:

1. 2600 milliliters of methylene chloride	5. 127 grams of anhydrous hydrazine
2. 1.5 grams of platinum foil	6. 2500 milliliters of 99% methanol
3. 1 gram of pure iodine	7. 300 grams of anhydrous sodium sulfate
4. 857 grams of dinitrogen pentoxide	

Reaction Equation:



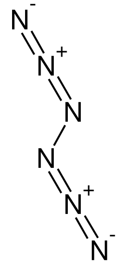
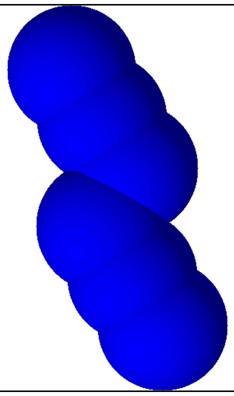
Preparation: Into a reflux apparatus equipped with a large 3-neck flask, two addition funnels, and motorized stirrer, pour in 500 milliliters of methylene chloride, followed by 1.5 grams of platinum foil, and then add in 1 gram of pure iodine. Thereafter, into the first addition funnel pour in 1/3rd of a mixture of 857 grams of dinitrogen pentoxide in 2100 milliliters of methylene chloride, and then pour into the second addition funnel, 127 grams of anhydrous hydrazine. Thereafter, turn on the stirrer, and then turn on the heating mantle and bring the temperature of the contents in the 3-neck flask up to 80 Celsius with rapid stirring. When the temperature of the contents in the 3-neck flask reaches 80 Celsius, begin a slow drip of the anhydrous hydrazine into the 3-neck flask, and at the same time, begin a rapid drip of the dinitrogen pentoxide/methylene chloride mixture into the 3-neck flask while maintaining a reflux at 80 Celsius under rapid stirring. When all the dinitrogen pentoxide/methylene chloride mixture in the addition funnel has been added, continue a slow drip of the anhydrous hydrazine in the second addition funnel into the 3-neck flask while maintaining a reflux of 80 Celsius under rapid stirring. Immediately thereafter, pour in another 1/3rd of the dinitrogen pentoxide/methylene chloride mixture into the first addition funnel, and then continue a rapid drip of the dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while continuing to heat the reaction mixture under reflux at 80 Celsius with constant stirring. By this time, all the anhydrous hydrazine should have been added. Now, pour the final 1/3rd of the dinitrogen pentoxide/methylene chloride mixture into the first addition funnel, and continue a rapid drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while maintaining the temperature of the reaction mixture at 80 Celsius under reflux with rapid stirring. After all the dinitrogen pentoxide/methylene chloride mixture in the addition funnel has been added, continue to reflux the reaction mixture at 80 Celsius under rapid stirring for 4 hours.



Setup for nitration of hydrazine.

Now, after 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature while continuing to stir the reaction mixture rapidly during the cool down period. Once the reaction mixture has reached room temperature, disassemble the apparatus, and then filter the reaction mixture using suction filtration to remove the platinum foil, and insoluble impurities. After the filtration process, take the filtered reaction mixture and place into a rotary evaporator, and evaporate-off the methylene chloride at 40 Celsius until a slightly yellowish liquid remains, and no more methylene chloride passes over. When this point has been achieved, stop the evaporation process, and recover the left over syrupy impure liquid product. Now, place this impure syrupy liquid into a large beaker, and then add in 2500 milliliters of 99% methanol, and then stir the combined mixture for 4 hours at room temperature. Note: use a motorized stirrer. After 4 hours, filter the entire mixture using suction filtration to remove any insoluble impurities. Thereafter, pour the filtered mixture into a large beaker filled with 5,000 milliliters of ice water, and then stir this new mixture for 1 hour. Note: the desired product tetranitrohydrazine will separate out as a liquid phase at the bottom of the beaker, and the methanol will dissolve in the ice water and form an upper phase. Finally, using a super large separatory funnel, or using a regular sized separatory funnel (and dividing the two phase mixture into portions at a time) place the two-phase mixture into the separatory funnel of your choice, and then carefully drain-off the bottom liquid phase. Once all the bottom liquid phase has been recovered, pour it into a fresh clean large beaker, and then add in 300 grams of anhydrous sodium sulfate to absorb moisture, and then stir the entire mixture for 1 hour. After 1 hour, filter-off the insoluble sodium sulfate, and then recover the desired liquid product and store in an amber glass bottle until use.

Theoretical Preparation 20: hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide; Hexazide; HTDD

 hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = N_6
Formula Weight = 84.0402
Composition = N(100.00%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 84.018444 Da
Nominal Mass = 84 Da
Average Mass = 84.0402 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6540	Non-flammable; does not ignite when mixed with reducing agents.	Non-sensitive to shock, heat, percussion, or friction. Very stable. Requires a significant blasting cap or detonator for initiation. A secondary explosive.	Very chemically inert. Does not react with oxidizing agents or reducing agents.	Transparent needle like crystals, white powder or granules. Melting point: 67 Celsius; boiling point: 309 Celsius under reduced pressure. Explodes when rapidly heated to 460 Celsius, Insoluble in water, and most organic solvents, but slightly soluble in liquid ammonia. Warning! This

				compound is toxic

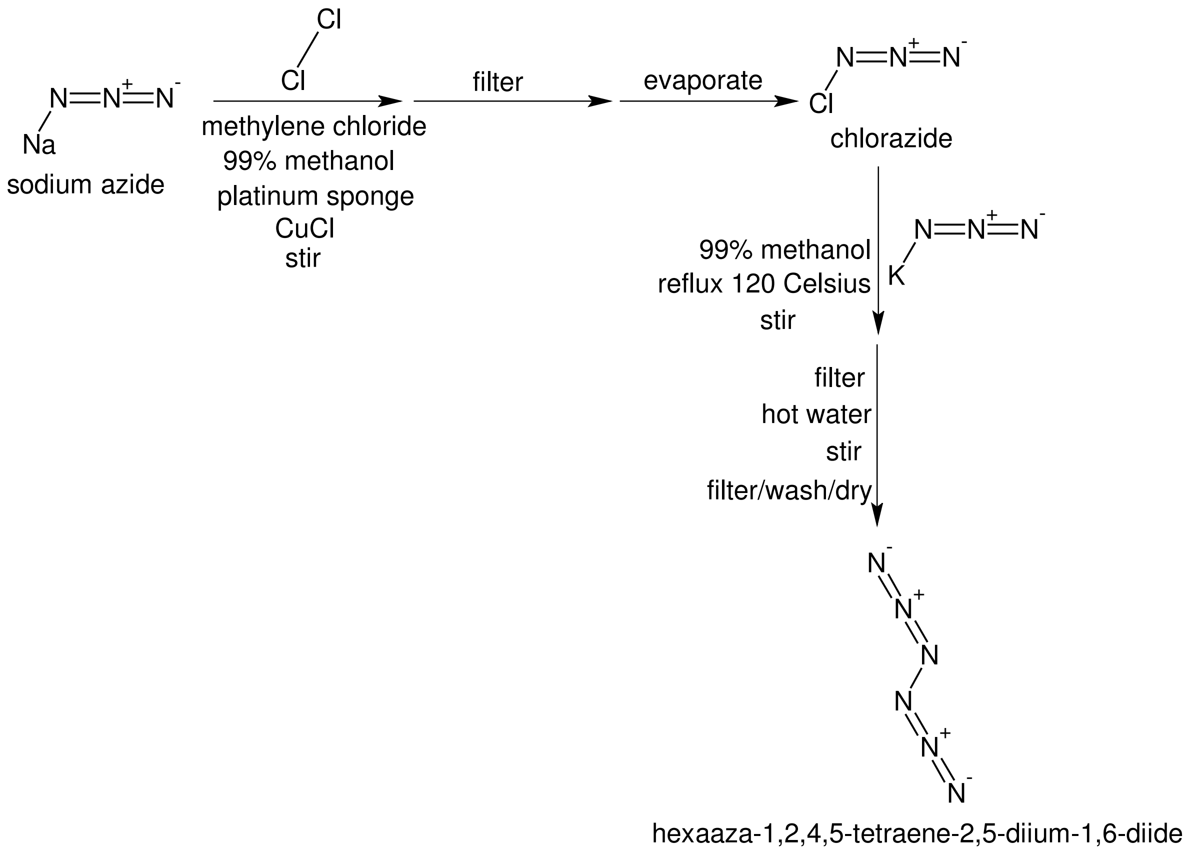
Estimated Compound summary: hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide is an amazing explosive that is 100% nitrogen. The explosive is a one of its kind, and has remarkable stability and physical properties. It can be stored for years without fear of decomposition or premature detonation; it does not require inert fillers for stability. hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide can be used when dead pressed into a variety of explosive munitions, or it can be melted down and cast into molds for shape charges, missile warheads, land mines, or torpedoes. hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide has limited use in drop bombs as cheaper explosives like TNT, RDX, are more readily available and easier of manufacture. The explosive is very chemically inert, and does not react with strong oxidizers even potassium permanganate, and it is non-reactive to the halogens, with the exception of fluorine gas, where it explodes on contact. Because of its amazing 100% nitrogen gas ratio, its explosiveness can be used in high performance gun propellants of the double-based type; however, because of its chemical inertness it cannot be used as a rocket fuel. hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide does not ignite when mixed with reducing agents including powdered metals, and the explosive has neither reducing, nor oxidizing properties, and is considered a neutral explosive that is only activated when subjected to extreme shock from a blasting cap or detonator; in most cases it requires a large blasting cap or detonator for initiation. hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide has a relatively low melting point, but it cannot be melted down and mixed with other molten explosives due to its lack of solubility. It is insoluble in molten explosives like TNT, RDX, HMX, PETN, and others. hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide can only be used by itself, and not mixed with other explosives.

Safety Info: Wear gloves, and avoid skin contact, ingestion, and inhalation of sodium azide and potassium azide, which are both highly toxic. Use proper ventilation and avoid inhalation of chlorine gas, which is toxic. Wear gloves, and avoid ingestion of methanol, which is toxic.

Materials:

1. 1950 milliliters of 99% methylene chloride	5. 4000 milliliters of 99% methanol
2. 5 grams of platinum sponge	6. 212 grams of chlorine gas
3. 50 grams of copper-I-chloride	7. 244 grams of potassium azide
4. 195 grams of sodium azide	

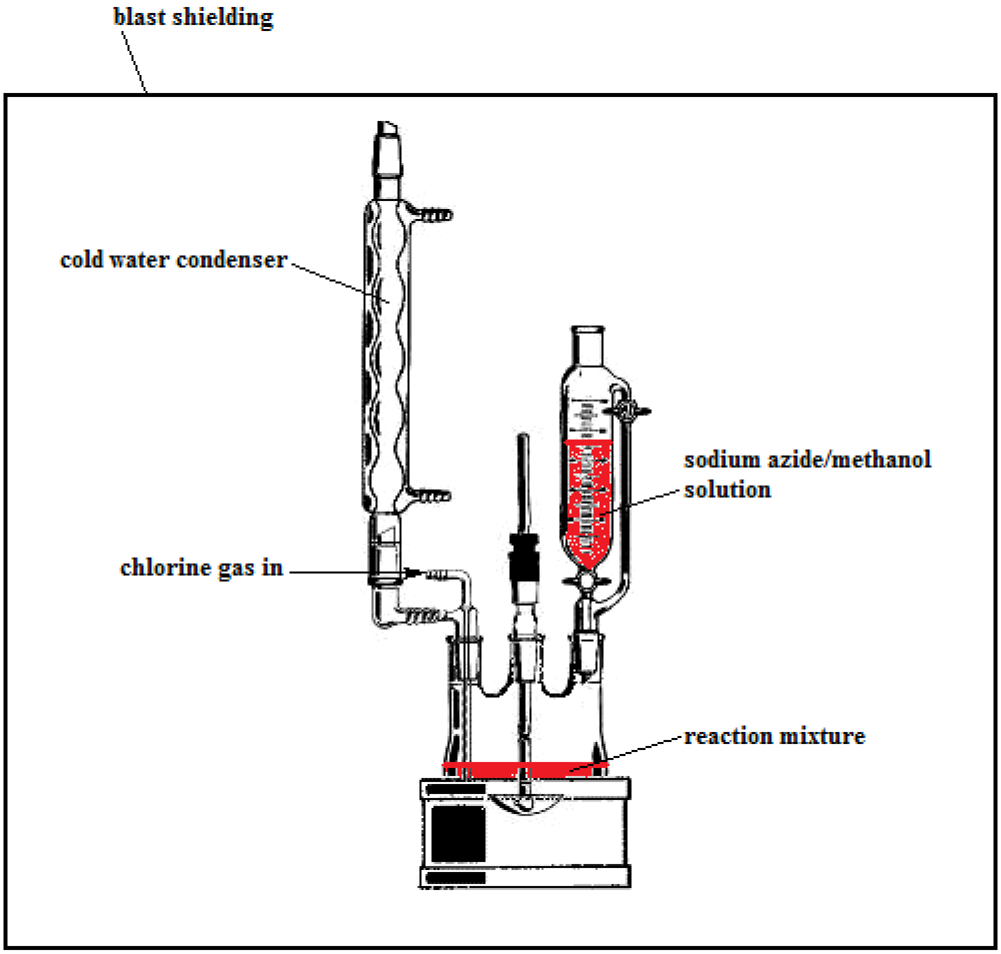
Reaction Equation:



Preparation:

Step 1: Preparation of chlorazide (3-chlorotriaza-1,2-dien-2-ium-1-ide)

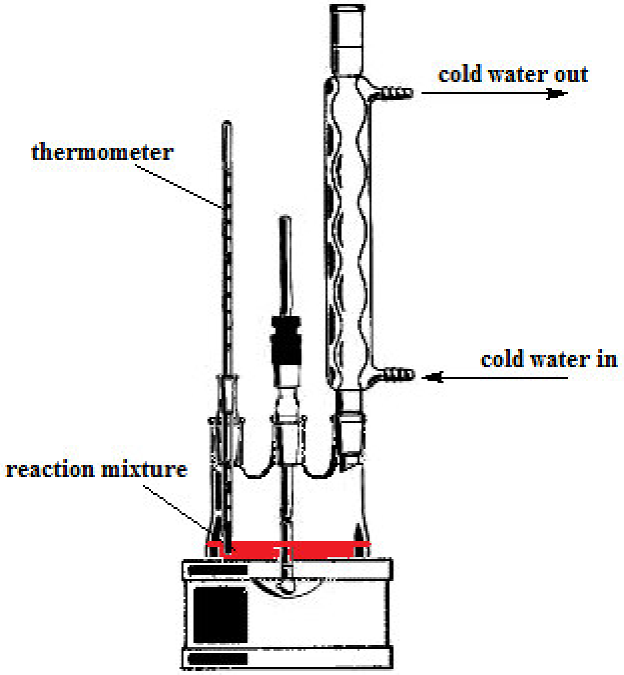
Warning! Use proper blast shielding when carrying out this procedure as chlorazide is very explosive. Into an apparatus as illustrated below, place 1500 milliliters of 99% methylene chloride, followed by 5 grams of platinum sponge, followed by 50 grams of copper-I-chloride, and then prepare a solution by using a clean beaker by dissolving 195 grams of sodium azide into 2500 milliliters of 99% methanol, and then once the sodium azide has dissolved, quickly place 1/3rd of this sodium azide/methanol solution into the addition funnel. Immediately thereafter turn on the heating mantle to 60 Celsius, and when its temperature has reached 60 Celsius, begin to bubble in chlorine gas at a flow rate of 200 mg per/minute. Immediately thereafter, begin a moderate drip of the sodium azide/methanol solution into the reaction mixture while rapidly stirring the reaction mixture and maintaining its temperature around 60 Celsius. Note: Continue to bubble in dry chlorine gas throughout this entire procedure until a total of 212 grams of chlorine gas has been added. After the sodium azide/alcohol mixture has been added from the addition funnel, pour in another 1/3rd of the sodium azide/methanol mixture into the addition funnel, and then continue a moderate drip of the sodium azide/methanol mixture into the reaction mixture while continuing to bubble in chlorine gas while rapidly stirring the reaction mixture and maintaining its temperature around 60 Celsius under mild reflux. After the sodium azide/methanol mixture from the addition funnel has been added, immediately pour in the last 1/3rd of the sodium azide/methanol solution into the addition funnel, and then continue a slow drip of this sodium azide/methanol solution into the reaction mixture while continuing to bubble in dry chlorine gas, and heating the reaction mixture at 60 Celsius under rapid stirring. After all the sodium azide/methanol solution has been added to the reaction mixture, continue to bubble in the chlorine gas while maintaining the reaction mixtures temperature around 60 Celsius with rapid stirring for 4 hours. Note: before this 4-hour period has been obtained all the chlorine gas would have been bubbled into the reaction mixture. After 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature with constant stirring. When the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter the reaction mixture to remove the insoluble copper-I-chloride, platinum sponge, and then insoluble by product sodium chloride. Now, place the filtered reaction mixture into a rotary evaporator and evaporate-off the methanol and methylene chloride under high vacuum and at a temperature of 50 Celsius until dry solid remains. When dry solid remains, stop the evaporation process, allow the remaining left over residue to cool to room temperature, and then recover this left over residue from the rotary evaporator, and store in a dessicator over phosphorus pentoxide until use. Note: chlorazide is hydrolyzed by water forming hydrogen chloride and the explosive hydrazoic acid, so use caution.



Setup for chlorination of sodium azide.

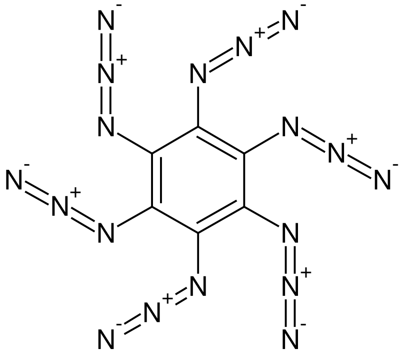
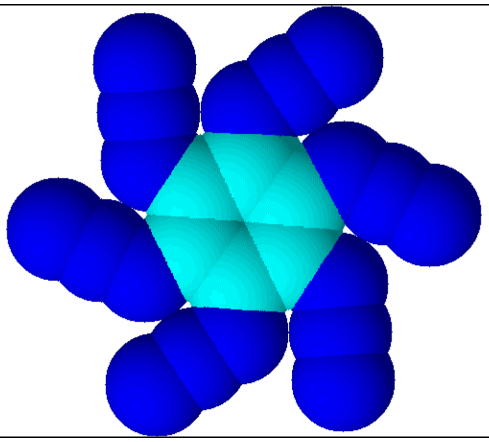
Step 2: Preparation of hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide

Into an apparatus as illustrated below, pour in 1500 milliliters of 99% methanol, followed by the chlorazide obtained from step 1. Thereafter add in 244 grams of potassium azide all in one portion, and then begin to stir the entire reaction mixture. Then immediately turn on the heating mantle, and heat the reaction mixture to 120 Celsius under reflux. When the temperature of the reaction mixture reaches 120 Celsius under reflux, continue to reflux and rapidly stir the reaction mixture for 6 hours. After 6 hours, remove the heat source, and then allow the reaction mixture to cool to room temperature. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter the reaction mixture using suction filtration to recover the insoluble product of hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide, along with the insoluble by-product of potassium chloride. Now, take this filtered-off mass, and place it into a large beaker containing 2500 milliliters of hot water, and then stir this mixture for 4 hours until its temperature reaches room temperature. Immediately thereafter, filter-off the insoluble product of hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide, wash with three 150-milliliter portions of methylene chloride, then with three 150-milliliter portions of hot water, and then suction dry as best as possible. Thereafter recover the washed product of hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide, and bake in an oven at 50 Celsius for 4 hours. After 4 hours, turn off the oven, and allow the dried product of hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide to cool to room temperature. Once it has, recover it from the oven, and then store in a glass container in a cool dry place until use.



Setup for preparation of hexaaza-1,2,4,5-tetraene-2,5-diium-1,6-diide.

Theoretical Preparation 21: hexaazidobenzene; HAAB; 6-Azide

 hexaazidobenzene	
Expanded structure	3D structure

Molecular data:

Molecular Formula = C_6N_{18}
Formula Weight = 324.1848
Composition = C(22.23%) N(77.77%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 324.055332 Da
Nominal Mass = 324 Da
Average Mass = 324.1848 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
5190	Non-flammable; but will burn when heated to above 250 Celsius and ignited with a flame.	Non-sensitive to heat, shock, percussion, friction, and pressure. Very stable. A secondary explosive. Requires blasting cap or detonator for initiation.	Non-reactive. Very chemically inert.	Colorless crystalline needles or white powder or granules. Melting point: 190 Celsius; boiling point: 345 Celsius under reduced pressure. Decomposes when heated to above 400 Celsius. Insoluble in water and most organic solvents; practically non-soluble in all known solvents.

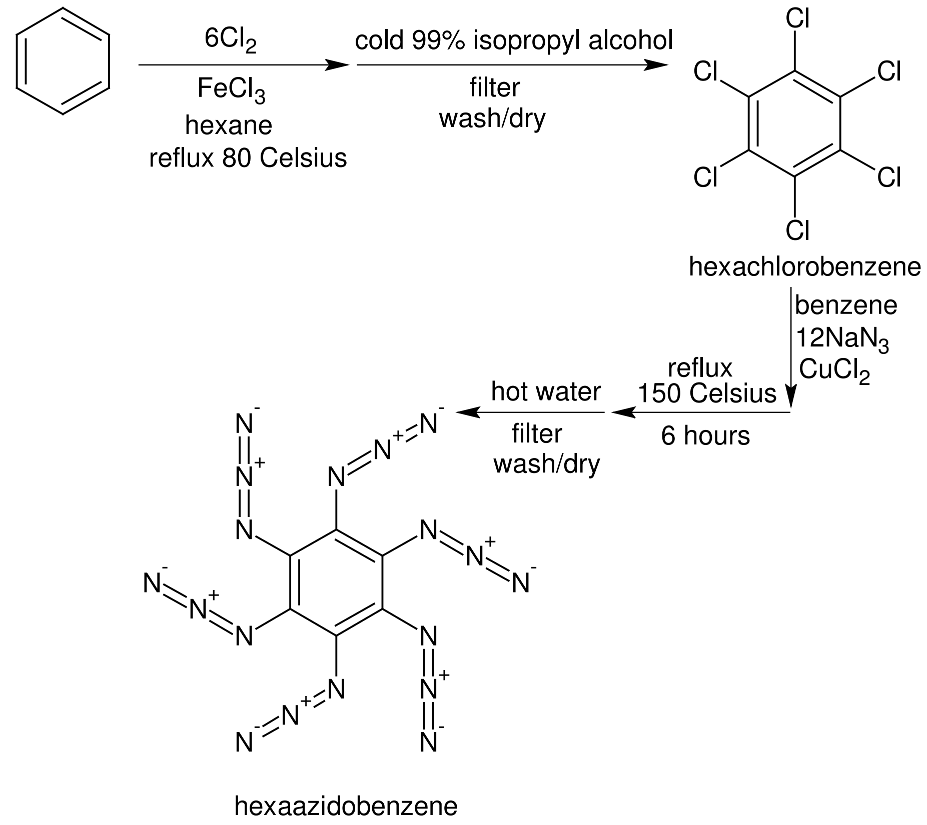
Estimated Compound summary: hexaazidobenzene is an interesting explosive that is very stable and inert. It shows great potential for use in explosives munitions such as high velocity projectiles, missile warheads, and space based rockets. Hexaazidobenzene is very chemically inert and shows no potential for use in high performance gun propellants or rocket fuels as it does not react nor burn when mixed with reducing agents and ignited. In some cases, hexaazidobenzene requires a significant blasting cap or detonator to be initiated especially when pure. Hexaazidobenzene can be melted down and casted into shape charges for use in commercial and military operations. Hexaazidobenzene cannot be melted down and mixed with other molten explosives due to its lack of solubility, and it is primarily used in pure form; however, it can be melted down, and ammonium nitrate added to form a special low velocity explosive for use in blasting soft and hard rock for mining operations. Hexaazidobenzene is very stable and chemically inert, and can be used with great potential in land mines, underwater munitions such as torpedoes, as it has excellent water resistance.

Safety Info: Extinguish all sources of ignition when handling hexane, which is highly volatile and flammable. Use proper ventilation and avoid inhalation, ingestion, and skin contact when handling benzene, which is a known carcinogen. Wear gloves and avoid skin contact, inhalation, or ingestion when handling sodium azide, which is highly toxic.

Materials:

1. 700 milliliters of dry hexane	6. 1750 milliliters of benzene
2. 126 grams of benzene	7. 10 grams of anhydrous copper-II-chloride
3. 10 grams of anhydrous ferric chloride	8. 816 grams of sodium azide
4. 343 grams of chlorine gas	9. 1500 milliliters of fresh 95% ethyl alcohol
5. 2400 milliliters of pre-chilled 99% isopropyl alcohol	10. 1500 milliliters of a 5% baking soda solution

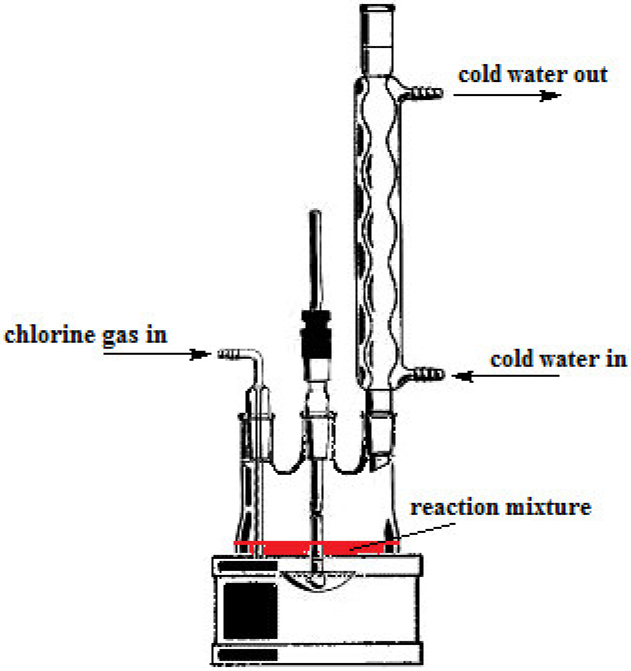
Reaction Equation:



Preparation:

Step 1: Preparation of hexachlorobenzene

Into an apparatus as illustrated below, place 700 milliliters of dry hexane, followed by 126 grams of benzene, and 10 grams of anhydrous ferric chloride. Thereafter, begin to stir the mixture using the motorized stirrer, and then turn on the heating mantle and bring the mixture to a reflux at 80 Celsius. When the mixtures temperature reaches 80 Celsius, begin to bubble in dry chlorine gas at a flow rate of 100 mg/per minute while refluxing the reaction mixture at 80 Celsius with rapid stirring. Continue to bubble in chlorine gas to the reaction mixture while refluxing it at 80 Celsius with rapid stirring until 343 grams of chlorine gas has been added. After all the chlorine gas has been added, continue to reflux the reaction mixture at 80 Celsius with rapid agitation for 3 hours. After 3 hours, turn off the heating mantle and allow the reaction mixture to come to room temperature. During this cool down period continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the reaction mixture into a large beaker filled with 1500 milliliters of pre-chilled 99% isopropyl alcohol (pre-chill in a freezer prior to using). Thereafter, rapidly stir this new mixture for 2 hours. After 2 hours, filter-off the insoluble desired product of hexachlorobenzene using suction filtration, wash with three 500-milliliter portions of a 5% baking soda solution, and then wash with three 300-milliliter portions of fresh pre-chilled 99% isopropyl alcohol, and then wash with three 500-milliliter portions of hot water, and then suction dry the desired product as best as possible, and then save for step 2.

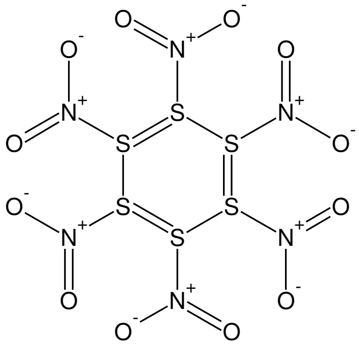
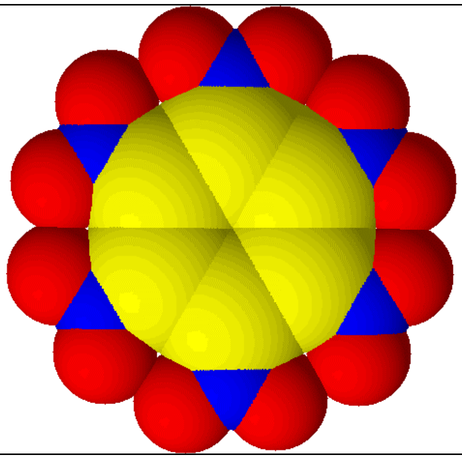


Setup for chlorination of benzene.

Step 2: Preparation of hexaazidobenzene

Into a standard reflux apparatus equipped with cold-water condenser, thermometer, and motorized stirrer, place 1750 milliliters of benzene, and then add in 10 grams of anhydrous copper-II-chloride. Thereafter, stir the mixture until the hexachlorobenzene dissolves in the benzene, and then add in 816 grams of sodium azide, and then turn on the heating mantle and begin a reflux at 150 Celsius with rapid stirring of the reaction mixture. Note: add in all the sodium azide at once then seal the apparatus and begin the reflux. Continue to reflux the reaction mixture at 150 Celsius with rapid agitation for 6 hours. After 6 hours, turn off the heating mantle and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during the cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a large beaker filled with 1500 milliliters of boiling water, and then stir this new hot mixture for 2 hours to dissolve the by-product sodium chloride. After 2 hours, filter the boiling hot mixture using suction filtration to recover the insoluble desired product of hexaazidobenzene, and then wash this filtered-off desired product with three 500-milliliter portions of fresh 95% ethyl alcohol, and then wash with three 500-milliliter portions of hot water. And then suction dry the washed product as best as possible. Finally, place the desired product of hexaazidobenzene in an oven, and bake at 100 Celsius for 6 hours. After 6 hours, turn off the oven, and allow the desired product of hexaazidobenzene to cool to room temperature. Then store the desired product in any desired container until use.

Theoretical Preparation 22: 1,2,3,4,5,6-hexanitro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine; Nitro hexathiine; Gamma-HNH

 1,2,3,4,5,6-hexanitro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $N_6O_{12}S_6$
Formula Weight = 468.423
Composition = N(17.94%) O(40.99%) S(41.07%)
Molar Refractivity = 81.51 ± 0.5 cm^3
Molar Volume = 107.4 ± 7.0 cm^3
Parachor = 632.2 ± 8.0 cm^3
Index of Refraction = 3.231 ± 0.05
Surface Tension = 1200.4 ± 7.0 dyne/cm
Density = 4.36 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 32.31 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 467.789839 Da
Nominal Mass = 468 Da
Average Mass = 468.423 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7156; estimated	Flammable solid; burns emitting toxic fumes of nitrogen oxides and sulfur oxides. Ignites violently when mixed with reducing agents, especially powdered aluminum. Mixtures with red phosphorus have a tendency to explode when ignited.	Mostly stable at room temperature but has a tendency to ignite under rapid friction. Non-sensitive to shock, and percussion. A secondary explosive. Requires blasting cap or detonator for initiation.	Generally non-reactive; reacts with strong oxidizing agents, halogens, and explodes when treated with fluorine. Decomposes slowly when added to boiling water.	Yellowish-red amorphous solid; Melting point: 89 Celsius; boiling point: decomposes when heated to 200 Celsius. Insoluble in water, and slowly hydrolyzed by it. Moderate solubility in carbon disulfide, but relatively insoluble in most organic

				solvents.

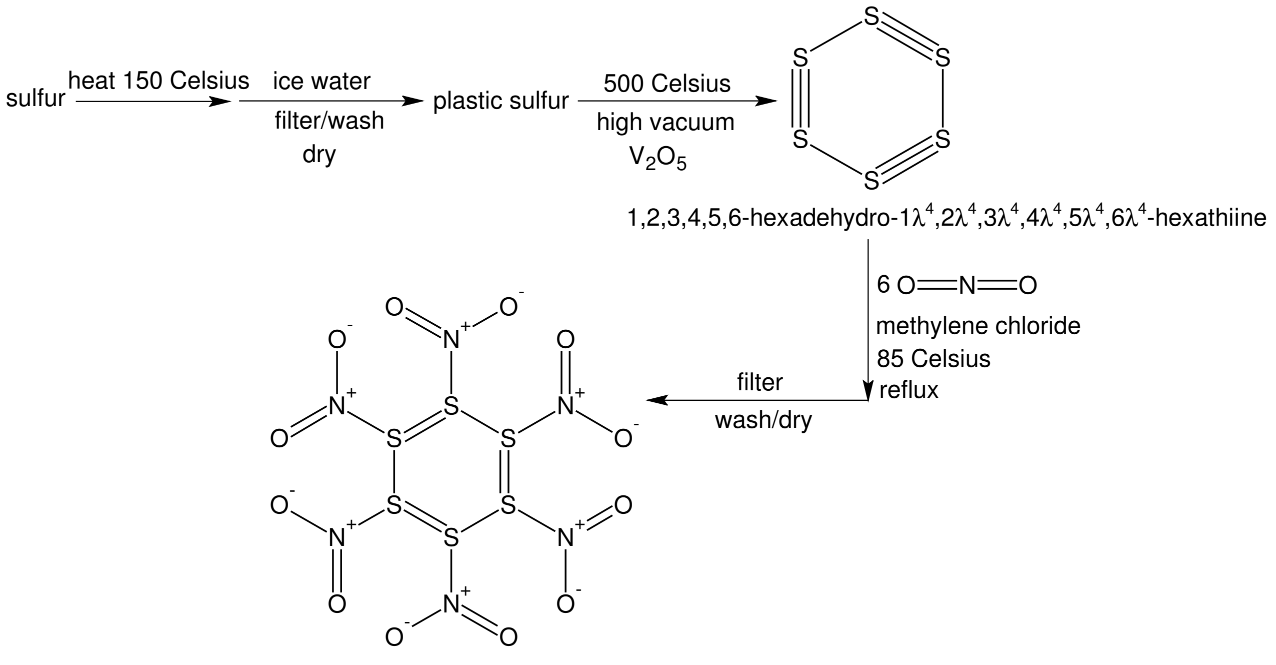
Estimated Compound summary: 1,2,3,4,5,6-hexanitro-1λ^4,2λ^4,3λ^4,4λ^4,5λ^4,6λ^4-hexathiine is an interesting cyclic sulfur based high explosive that has a relatively low melting point. This low melting point makes it well suitable for melting and casting into molds such as shape charges, mortars shells, artillery shells, missile warheads, and pretty much any other desired means. 1,2,3,4,5,6-hexanitro-1λ^4,2λ^4,3λ^4,4λ^4,5λ^4,6λ^4-hexathiine forms explosive mixtures with reducing agents, and hence can be used in making modified gun powders and for making a wide variety of pyrotechnics for many different uses. It can also be used in making fireworks for making bursting charges for aerial displays. 1,2,3,4,5,6-hexanitro-1λ^4,2λ^4,3λ^4,4λ^4,5λ^4,6λ^4-hexathiine can also be used to make high performance rocket fuels when melted and mixed with powdered aluminum or any powdered metal or strong reducing agents and epoxy, and also in making double based high performance gun propellants.

Safety Info: Extinguish all flames and sources of ignition before using 95% ethyl alcohol and 99% isopropyl alcohol both of which are flammable. Use extreme caution and use maximum ventilation when handling nitrogen dioxide, which is highly toxic; avoid inhalation.

Materials:

1. 200 grams of flours of sulfur	5. Nitrogen gas from tank
2. 50 grams of vanadium pentoxide	6. 287 grams of dry nitrogen dioxide gas
3. 900 milliliters of 95% ethyl alcohol	7. 1500 milliliters of pre chilled 99% isopropyl alcohol
4. 1800 milliliters of methylene chloride	

Reaction Equation:

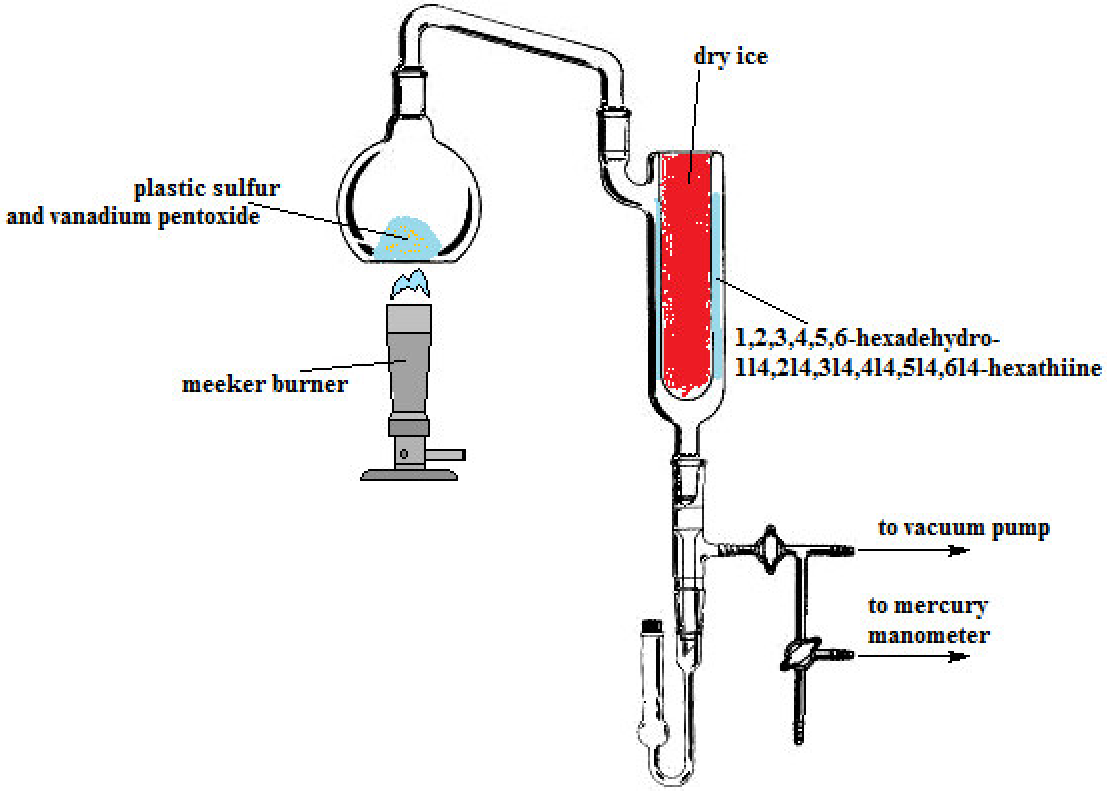


1,2,3,4,5,6-hexanitro-1λ^4,2λ^4,3λ^4,4λ^4,5λ^4,6λ^4-hexathiine

Preparation:

Step 1: Preparation of 1,2,3,4,5,6-hexadehydro-1λ^4,2λ^4,3λ^4,4λ^4,5λ^4,6λ^4-hexathiine

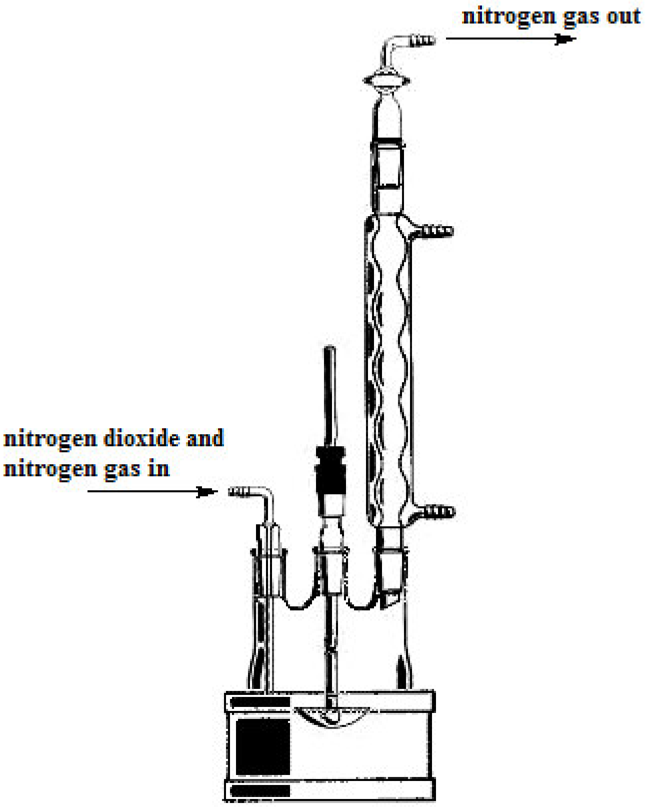
Into a suitable sized beaker equipped with motorized stirrer, and hot plate, place 200 grams of flours of sulfur. Thereafter, turn on the hot plate, and heat to 150 Celsius. When the temperature rises above 100 Celsius the sulfur will melt. Continue to stir the molten sulfur at 150 Celsius for 1 hour. After 1 hour, quickly pour the molten sulfur into a beaker containing 1500 milliliters of ice water. At this point the sulfur will solidify into plastic sulfur. After pouring in the molten sulfur to the ice water, allow the plastic sulfur to remain in the ice water until the ice melts. Once this point has been achieved, filter-off the insoluble plastic sulfur using suction filtration, and then wash with three 150-millilter portions of hot water, and then suction dry as best as possible. Now, into an apparatus as illustrated below, place the plastic sulfur into the reaction flask, and then add in 50 grams of vanadium pentoxide. Then turn on the meeker burner, and heat the plastic sulfur to 500 Celsius under a high vacuum. Continue to heat the plastic sulfur for 6 hours at 150 Celsius under high vacuum. During this time, the plastic sulfur will be converted to 1,2,3,4,5,6-hexadehydro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine, which will solidify on the outer walls of the dry ice condenser. After 6 hours, turn off the meeker burner, and allow the apparatus to cool to room temperature. Note: the dry ice will have to be replaced periodically as it will evaporate during the 6-hour heating process. Now, disassemble the apparatus, remove any dry ice that remains in the dry ice condenser and then remove the dry ice condenser and using any desired means, scrape off the 1,2,3,4,5,6-hexadehydro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine from the outer walls of the dry ice condenser, and then place this scraped-off mass into a Buchner funnel, and under suction, wash with three 300-milliliter portions of 95% ethyl alcohol, and then suction dry the desired 1,2,3,4,5,6-hexadehydro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine product and save for step 2.



Set-up for the formation of 1,2,3,4,5,6-hexadehydro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine.

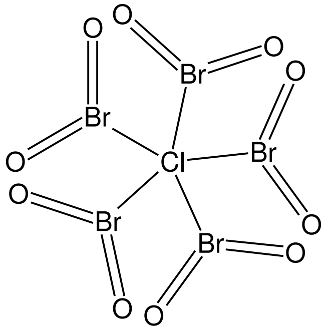
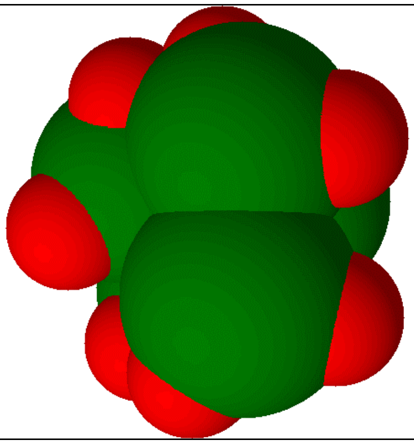
Step 2: Preparation of 1,2,3,4,5,6-hexanitro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine

Into an apparatus as illustrated below, place the dried product from step 1 into the 3-neck flask, and then add in 750 milliliters of methylene chloride, and then begin a nitrogen gas purge at a flow rate of 500 mg per/minute to sweep out any oxygen. Thereafter, turn on the heating mantle and bring the contents in the 3-neck flask to come to high reflux by heating the contents to 85 Celsius with rapid stirring. When the temperature of the reaction mixture in the 3-neck flask reaches 85 Celsius, begin to bubble in dry nitrogen dioxide gas at a flow rate of 150 mg per/minute along with dry nitrogen at a flow rate of 500 mg per/minute while refluxing the reaction mixture at 85 Celsius under rapid stirring. Continue to bubble in the nitrogen dioxide gas until 287 grams of dry nitrogen dioxide gas has been added. Maintain a high reflux at 85 Celsius with rapid stirring during the entire addition of the nitrogen dioxide gas. After all the nitrogen dioxide gas has been added, continue to bubble in dry nitrogen gas at a flow rate of 500 mg per/minute while maintaining a high reflux at 85 Celsius under rapid stirring for 4 hours. After 4 hours, remove the nitrogen gas purge, and then turn off the heating mantle and allow the reaction mixture to cool to room temperature. Once this point has been achieved, disassemble the apparatus and filter-off the insoluble desired product of 1,2,3,4,5,6-hexanitro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine using suction filtration, wash with three 350-milliliter portions of fresh methylene chloride, followed by three 500-milliliter portions of pre chilled 99% isopropyl alcohol, and then suction dry as best as possible.



Setup for the nitration of 1,2,3,4,5,6-hexadehydro-$1\lambda^4,2\lambda^4,3\lambda^4,4\lambda^4,5\lambda^4,6\lambda^4$-hexathiine.

Theoretical Preparation 23: pentakis(dioxidobromanyl)-λ^5-chlorane; Chlorane; pentabromate chloride; PDDBC

 pentakis(dioxidobromanyl)-λ^5-chlorane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = Br_5ClO_{10}
Formula Weight = 594.967
Composition = Br(67.15%) Cl(5.96%) O(26.89%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available

Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 589.509649 Da
Nominal Mass = 590 Da
Average Mass = 594.967 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
5145	Non-flammable; ignites and burns violently when mixed with reducing agents or powdered metals and touched by a spark or flame.	Stable at room temperature. Hydrolyzes by water (half life 45 minutes) A secondary explosive. Requires blasting cap or detonator for initiation.	Reacts with water forming bromic acid, and hydrogen chloride. Explodes in contact with fluorine. Decomposes when heated above 90 Celsius. Reacts with alcohols and glycols introducing bromite groups.	Orange-lemon cubic crystals. Melting point: decomposes; Boiling point: decomposes. Insoluble in water and hydrolyzed by it. Insoluble in all know solvents.

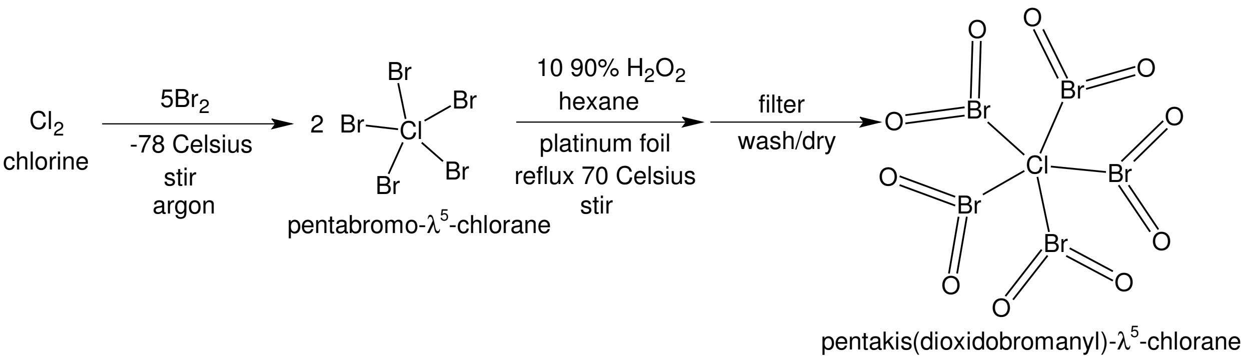
Estimated Compound summary: pentakis(dioxidobromanyl)-λ^5-chlorane is a fascinating new explosive with a central chlorine atom bonded to bromic groups. The compound has some draw backs in the fact that it cannot be melted down and casted into molds, and the only method for its use in explosive munitions is by dead pressing it into mortar shells, artillery rounds, and drop bombs under high pressure; however, pentakis(dioxidobromanyl)-λ^5-chlorane does show potential use in pyrotechnics for a variety of uses including flares, bursting charges for fireworks, making other fireworks such as crackling effects in shells, cakes, ect., manufacturing high velocity gun propellants, signals, matches, and many other pyrotechnic devices. Although, it cannot be used to make rocket propellants, as its decomposition point often leads to premature explosion of the rocket shortly after firing. pentakis(dioxidobromanyl)-λ^5-chlorane shows remarkable use for making propellant charges for heaving shells such as high explosive mortar rounds, and mortar shells in fireworks. pentakis(dioxidobromanyl)-λ^5-chlorane can also be dead pressed into card board tubes and used in demolition charges or for making explosive charges for blasting soft or hard rock. One interesting attribute that pentakis(dioxidobromanyl)-λ^5-chlorane has, is when detonated it forms explosive gases such as chlorine monoxide chlorine dioxide, and bromine oxides, which often detonate spontaneously after the original detonation, making it even more potent as a high explosive.

Safety Info: Use good ventilation, and wear gloves and face mask when handling liquid bromine, which is very volatile and emits toxic and irritating fumes; use caution and avoid skin contact, ingestion, and inhalation. Use proper ventilation when handling chlorine gas, and avoid inhalation of the toxic gas. Extinguish all flames and sources of ignition when handling hexane, which is highly flammable. Use care when handling 90% hydrogen peroxide, as it is a strong oxidizing agent; keep out contact with finely divided metals, and water soluble metal salts.

Materials:

1. 1,149 grams of liquid bromine	4. 5 grams of platinum foil
2. 102 grams of chlorine gas	5. 550 grams of 90% hydrogen peroxide
3. 750 milliliters of dry hexane	6. 2250 milliliters of methylene chloride

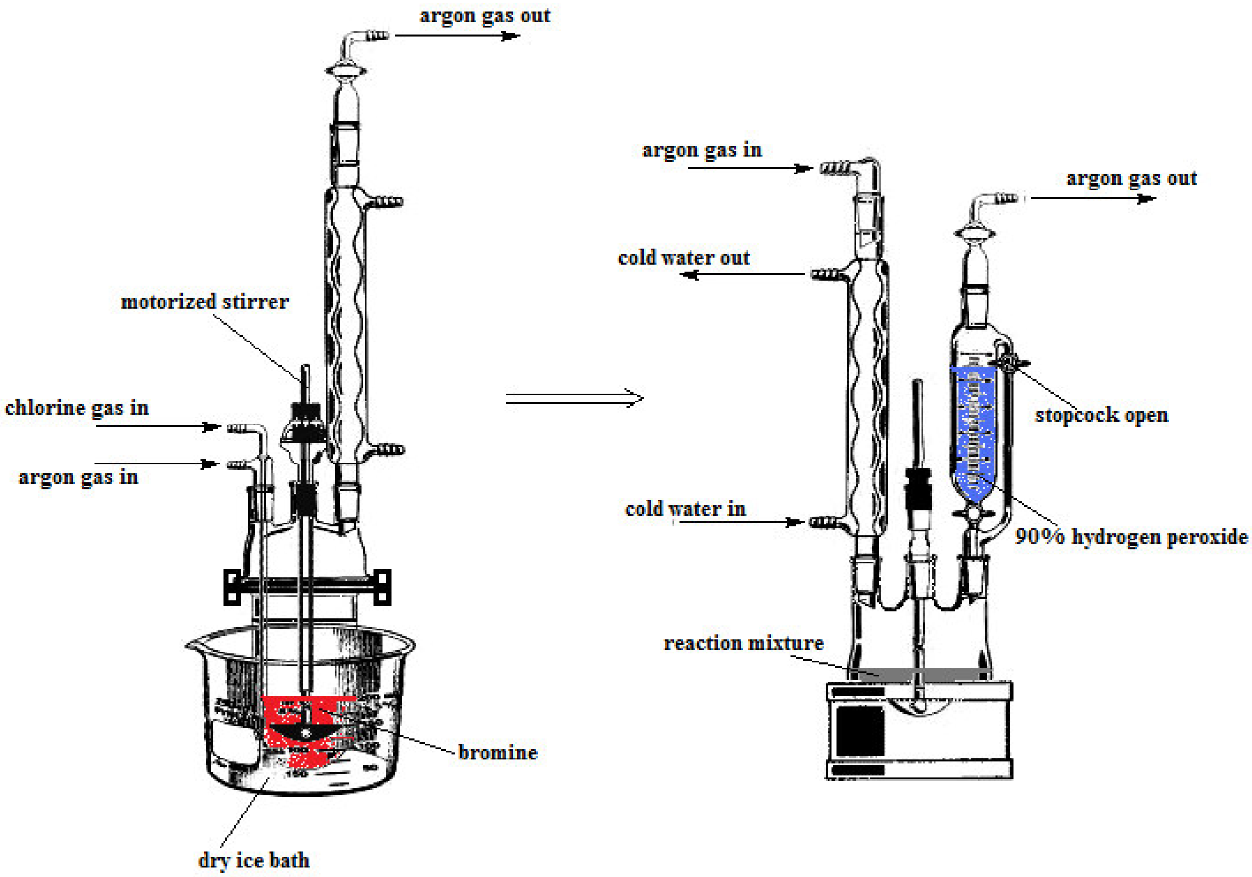
Reaction Equation:



Preparation:

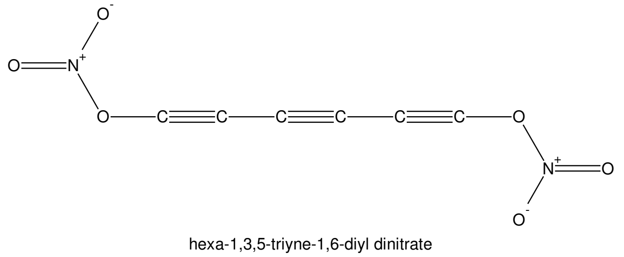
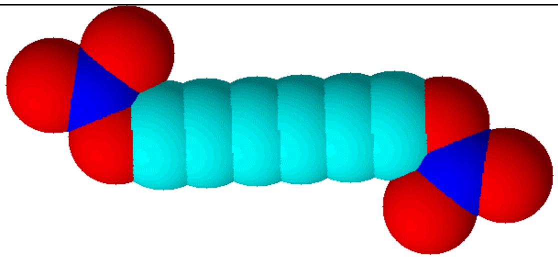
Step 1 and 2 combined: preparation of pentabromo-λ^5-chlorane and pentakis(dioxidobromanyl)-λ^5-chlorane

Into the left illustration as illustrated below, purge the apparatus with argon gas, and then place 1,149 grams of liquid bromine into the reaction vessel. Thereafter, pour dry ice into the container surrounding the reaction vessel, and chill the bromine to –78 Celsius. When the bromines temperature reaches –78 Celsius, begin to bubble into the bromine, dry chlorine gas at a flow rate of 150 mg per/minute while rapidly stirring the contents in the reaction vessel. Continue to bubble in dry chlorine gas into the bromine reaction mixture until 102 grams of chlorine gas has been bubbled into the reaction vessel. Continue to purge the apparatus with argon during the whole chlorine addition process. Note: maintain the temperature of the reaction mixture in the reaction vessel at around –78 Celsius with rapid stirring at all times—second note: the dry ice will have to be replaced with fresh dry ice periodically during the whole process. After all of the chlorine gas has been added, continue to stir the reaction mixture in the reaction vessel at –78 Celsius for 4 hours with rapid stirring, and continue the argon purge during this whole process. After 4 hours, remove the argon gas purge, and then quickly dissemble the apparatus, and then quickly assemble the apparatus as illustrated to the right of the illustration shown below. Immediately thereafter, scrape out the desired liquid slush mass of pentabromo-λ^5-chlorane from the reaction vessel, and place it into the 3-neck flask, followed by 750 milliliters of dry hexane, followed by 5 grams of platinum foil. Thereafter, bring the hexane to a gentle reflux by heating it to 70 Celsius using the heating mantle, and when its temperature reaches 70 Celsius, quickly pour into the addition funnel, 550 grams of 90% hydrogen peroxide, and then begin a moderate drip of this hydrogen peroxide into the reaction mixture. During the addition of the hydrogen peroxide, continue to reflux the reaction mixture at 70 Celsius with rapid stirring. After all the hydrogen peroxide has been added, continue to reflux the reaction mixture at 70 Celsius with rapid stirring for 6 hours. After 6 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Once it has, disassemble the apparatus, and then filter-off the insoluble desired product of pentakis(dioxidobromanyl)-λ^5-chlorane using suction filtration. Note: be sure to recover the platinum foil from the filter cake. Finally, wash the filtered-off desired product of pentakis(dioxidobromanyl)-λ^5-chlorane with three-750 milliliter portions of fresh methylene chloride, and then suction dry as best as possible. Thereafter, place the desired dried product of pentakis(dioxidobromanyl)-λ^5-chlorane into an oven, and carefully bake at 50 Celsius for 6 hours. After 6 hours, turn off oven, and allow the desired product of pentakis(dioxidobromanyl)-λ^5-chlorane to cool to room temperature, then store it in a dessicator over phosphorus pentoxide until use.



Double setup for the preparation of pentakis(dioxidobromanyl)-λ^5-chlorane.

Theoretical Preparation 24: hexa-1,3,5-triyne-1,6-diyl dinitrate; HTDD; poly acetylene dinitrate

 O O N O C C C C C C O N O O hexa-1,3,5-triyne-1,6-diyl dinitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6N_2O_6$
Formula Weight = 196.074
Composition = C(36.75%) N(14.29%) O(48.96%)
Molar Refractivity = 39.39 ± 0.3 cm^3
Molar Volume = 115.7 ± 3.0 cm^3
Parachor = 361.7 ± 4.0 cm^3
Index of Refraction = 1.596 ± 0.02
Surface Tension = 95.3 ± 3.0 dyne/cm
Density = 1.693 ± 0.06 g/cm^3
Dielectric Constant = Not available
Polarizability = 15.61 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 195.975636 Da

Nominal Mass = 196 Da
Average Mass = 196.074 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6700	Flammable solid; in absence of air burns with a reddish flame emitting nitrogen oxides and leaving a sooty residue; burns in air producing nitrogen oxides, and carbon oxides	Very stable; non-sensitive to shock, heat, friction, percussion, and pressure. A secondary explosive. Requires blasting cap or detonator for initiation.	Stable at room temperature and temperatures up to 400 Celsius. Detonates when rapidly heated to 500 Celsius. Reacts with halogens forming addition products; reacts with finely divided copper forming very sensitive explosives; other then that pretty much un-reactive.	White powder or granules; impure compound will have a grayish tint. Melting point 57 Celsius, Boiling point: 190 Celsius under reduced pressure. Insoluble in water, but moderately soluble in methylene chloride, acetone, alcohol, and hexane. Soluble in ether. Very soluble in concentrated sulfuric acid.

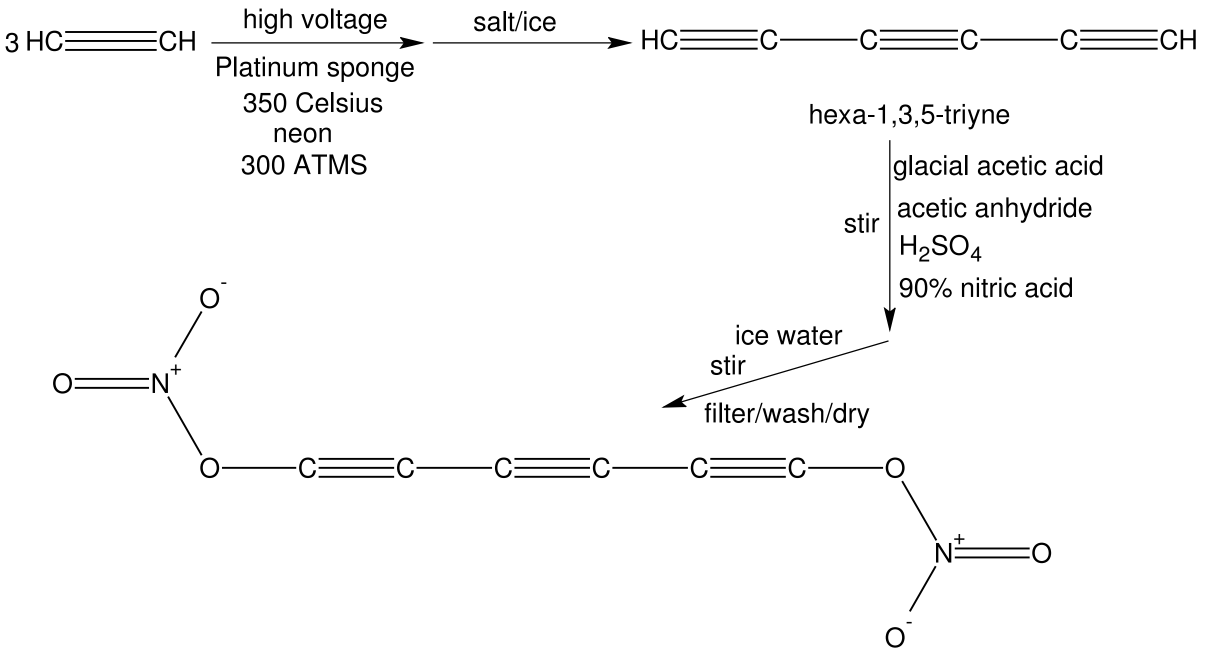
Estimated Compound summary: hexa-1,3,5-triyne-1,6-diyl dinitrate is a new and radical explosive in a line of new explosives of the poly acetylene class. The compound has a moderate detonating velocity making it less powerful then TNT, but it has a very low melting point and is soluble in many other molten explosives such as TNT, RDX, HMX, and many others; therefore it can be melted and used by-itself or alloyed with many other explosives to form very stable explosive compositions for use in making shape charges, mortar shells, artillery shells, high velocity projectiles, missile warheads, land mines, and torpedoes. hexa-1,3,5-triyne-1,6-diyl dinitrate is very water resistant and is perfect for making explosive munitions that will have to be exposed to poor weather conditions over prolonged periods of time. The explosive is also capable of forming composition for making high performance rocket fuels when mixed with powdered aluminum or solid strong reducing agents and cured with plasticizers or epoxies. One of the main uses of this compound is to make other high explosives that will be discussed later.

Safety Info: Take care when handling acetylene gas as it is highly flammable; extinguish all sources of ignition before using. Wear gloves and avoid skin contact when handling glacial acetic acid and acetic anhydride as they both can cause skin burns. Wear gloves and avoid skin contact when handling concentrated sulfuric acid, which is very corrosive and is capable of charring many substances. Use extreme care when handling 90% nitric acid, as it is highly volatile and emits toxic fumes of nitrogen oxides, avoid inhalation, and use maximum ventilation.

Materials:

1. Neon gas from tank	5. 750 milliliters of concentrated sulfuric acid
2. 203 grams of acetylene gas	6. 370 grams of 90% nitric acid
3. 100 milliliters of glacial acetic acid	7. 1500 milliliters of a 10% sodium carbonate solution
4. 100 milliliters of acetic anhydride	

Reaction Equation:

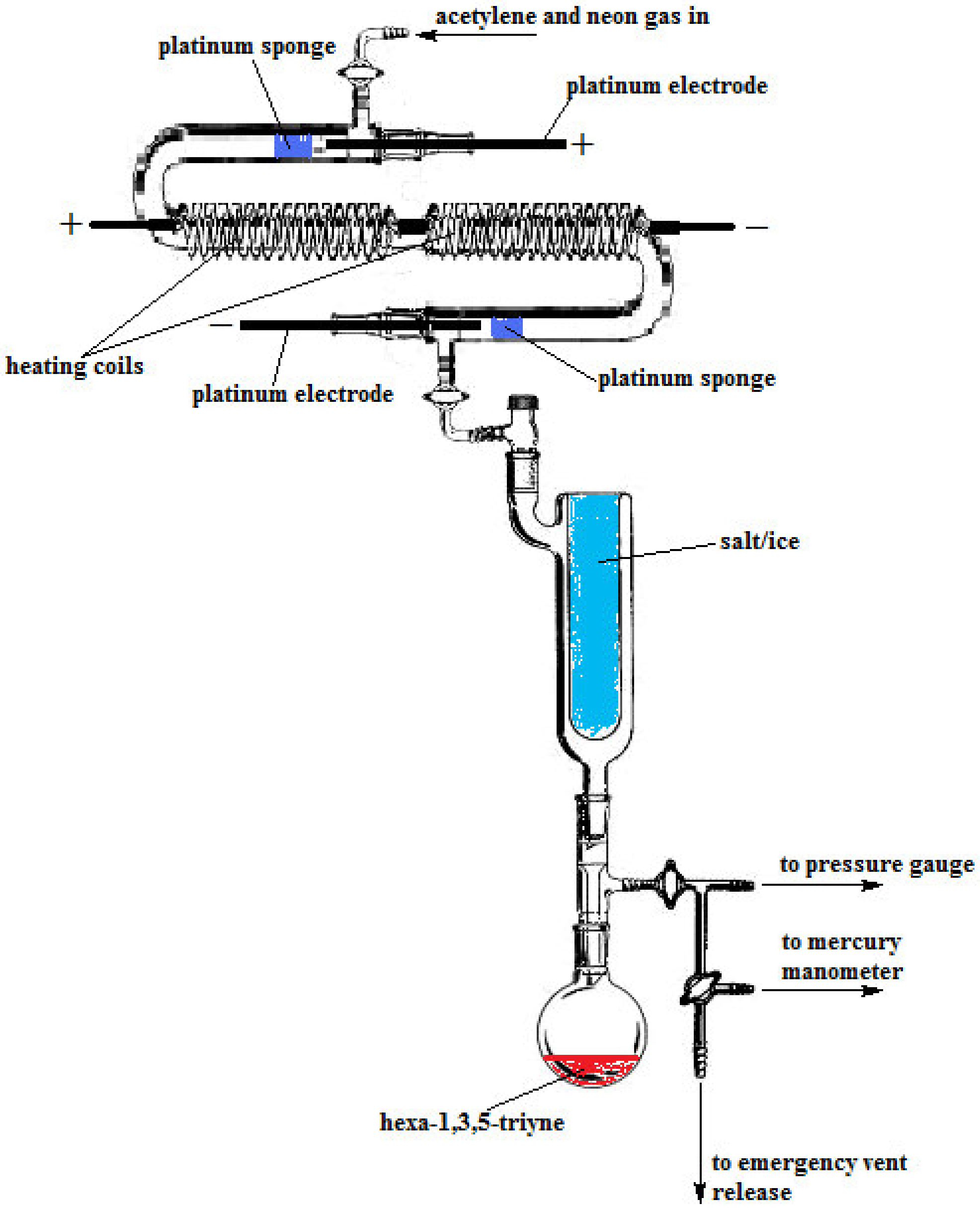


hexa-1,3,5-triyne-1,6-diyl dinitrate

Preparation:

Step 1: Preparation of hexa-1,3,5-triyne

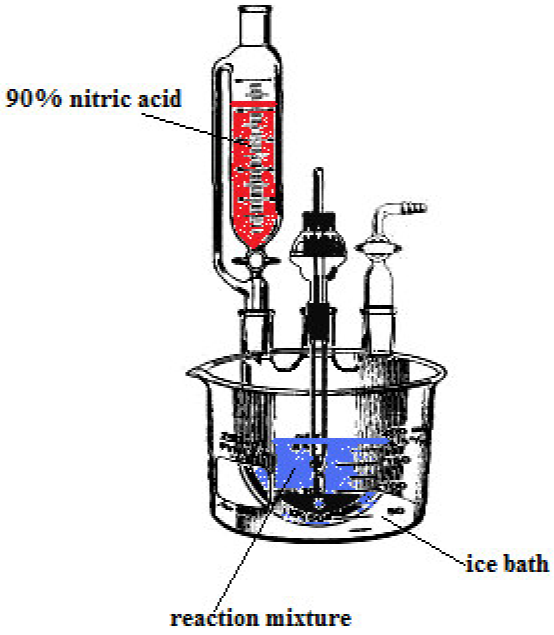
Into a specialized apparatus as illustrated below, begin a neon gas purge to raise the pressure inside the apparatus to 300 ATMS. Thereafter, turn on the power supply to the heating coils, and get the temperature of the heating coils to 350 Celsius. When the coils are at 350 Celsius, using extreme caution, and using a high voltage power supply, attach the negative lead clamp to the platinum cathode, and attach the positive clamp to the platinum anode. Thereafter turn on the power supply to 150,000 Volts, at 500 milliamps. Immediately thereafter, continue to purge the apparatus with neon gas, and then begin to pass in dry acetylene gas at a flow rate of 500 mg per/minute. While heating the heating coils to 350 Celsius and continuing the high voltage, continue to purge the apparatus with neon gas and continue to pass in acetylene gas at a flow rate of 500 mg per/minute until 203 grams of acetylene gas has been passed in. Note: the high voltage will cause the neon gas to glow a bright fluorescent green color inside the apparatus. Once 203 grams of acetylene gas has been passed in, turn off the high voltage power supply, and turn off the power source feeding the electric heating coils, and stop the neon gas purge. Note: during the process, the salt/ice will have to be replaced periodically to condense the desired product of hexa-1,3,5-triyne, which is a liquid at room temperature. Once the apparatus has cooled to room temperature, and all power supplies have been turned off, release the pressure gauge to bring the pressure in side the apparatus back to atmospheric pressure. Thereafter, disassemble the apparatus, and save the desired liquid product of hexa-1,3,5-triynen for step 2.



Setup for the preparation of hexa-1,3,5-triyne. Note: the upper portion of the apparatus is made of quartz/silica glassware.

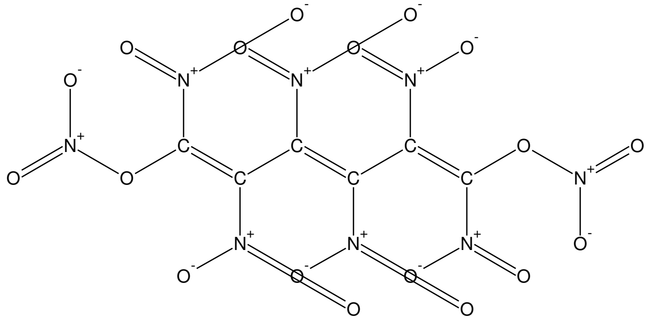
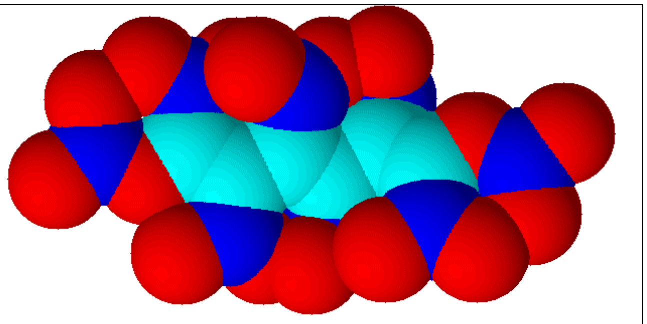
Step 2: Preparation of hexa-1,3,5-triyne-1,6-diyl dinitrate

Into an apparatus as illustrated below, place 100 milliliters of glacial acetic acid, followed by 100 milliliters of acetic anhydride, followed by 750 milliliters of concentrated sulfuric acid. Thereafter, pour in 370 grams of 90% nitric acid into the addition funnel. Thereafter, place the apparatus in an ice bath and chill the reaction mixture to 5 to 10 Celsius. When the reaction mixtures temperature reaches 5 to 10 Celsius, begin to the rapidly stir the reaction mixture, and then begin a slow drip of the 90% nitric acid into the reaction mixture. Continue to rapidly stir the reaction mixture and maintain its temperature around 5 to 10 Celsius under rapid stirring during the entire addition of the nitric acid. After the addition of the nitric acid, continue to rapidly stir the reaction mixture at a temperature around 5 to 10 Celsius under rapid stirring for 4 hours. Note: during the initial addition of the nitric acid, the ice bath will have to be replaced with fresh ice periodically during the process. After 4 hours, remove the ice bath, and then disassemble the apparatus, and then gently and slowly pour the entire reaction mixture into 2500 milliliters of ice water. Note: use caution as concentrated sulfuric acid generates allot of heat when added to water. After the reaction mixture has been completely added to the ice water, stir the entire mixture for 4 hours. After 4 hours, filter-off the insoluble desired product of hexa-1,3,5-triyne-1,6-diyl dinitrate using suction filtration, wash with three 500-milliliter portions of a 10% sodium carbonate solution, followed by three 500-milliliter portions of hot water, and then suction dry as best as possible. Finally, place the suction dried desired product onto a shallow pan and allow it to thoroughly dry at room temperature.



Simple setup for the nitration of hexa-1,3,5-triyne.

Theoretical Preparation 25: 1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate; HNHTDD; Hexanitro-Triene; Triene dinitrate

 1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6N_8O_{18}$
Formula Weight = 472.107
Composition = C(15.26%) N(23.73%) O(61.00%)
Molar Refractivity = 80.74 ± 0.3 cm^3
Molar Volume = 222.1 ± 3.0 cm^3
Parachor = 717.2 ± 4.0 cm^3
Index of Refraction = 1.647 ± 0.02
Surface Tension = 108.6 ± 3.0 dyne/cm
Density = 2.124 ± 0.06 g/cm^3
Dielectric Constant = Not available
Polarizability = 32.01 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 471.933055 Da
Nominal Mass = 472 Da
Average Mass = 472.107 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7145	Non-flammable. Will not ignite when heated to 600 Celsius, but decomposes violently producing nitrogen oxides and a sooty carbon residue.	Non-sensitive to shock, percussion, heat, pressure, and friction. Very stable. A secondary explosive. Requires blasting cap or detonator for initiation.	Generally non-reactive. Reacts with hydrogen halides forming addition products. Also reacts with halogens forming addition products; explodes in contact with fluorine.	White crystalline mass, powder, or granules. Melting point: 189 Celsius; Boiling point: 478 Celsius under reduced pressure. Begins to decompose when heated in air to 500 Celsius, and complete disintegration occurs when heated to 600 Celsius. Insoluble in water, and alcohol, but soluble in methylene chloride, acetone, hexane, toluene, and slightly soluble in ether.

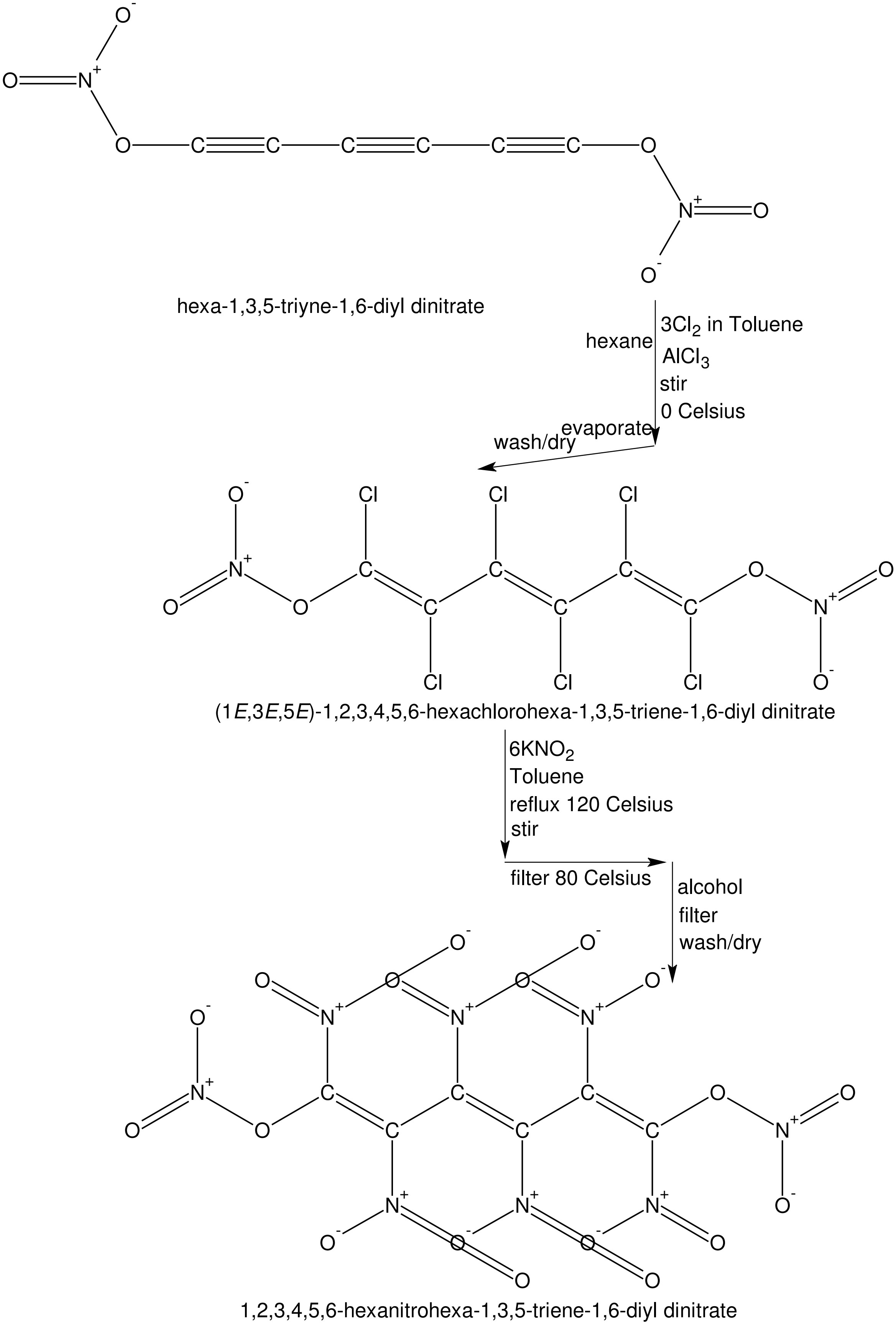
Estimated Compound summary: 1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate is a fascinating new explosive with tremendous power. Its remarkable high melting point makes it perfect for explosive munitions that are exposed to heat such as high velocity explosive shells, space based weapons, rockets, and artillery shells. The explosive is also very inert and resistant to chemical and bad weather attack, so it can be used in munitions for use in poor weather environments. 1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate shows remarkable use in grenades, land mines, and mortar shells. The explosive can also be easily melted down and casted into molds for use in making shape charges, or melted down and mixed with other explosives such as TNT, RDX, HMX, PETN, ect., for use in making very stable and environmentally stable explosive compositions. 1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate can also be used in making high performance gun propellants of the double based type, or used to make high performance rockets fuels when mixed with powdered metals and plasticizers and/or epoxy.

Safety Info: Extinguish all flames and sources of ignition before handling hexane, which is highly volatile and flammable. Wear gloves and avoid skin contact and contact with water when handling anhydrous aluminum chloride. Use proper ventilation and avoid inhalation when handling chlorine, which is toxic. Use proper ventilation and avoid skin contact and inhalation when handling toluene, which is a known carcinogen.

Materials:

1. 500 milliliters of dry hexane	5. 1094 grams of toluene
2. 10 grams of anhydrous aluminum chloride	6. 750 milliliters of toluene
3. 52 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2)	7. 136 grams of potassium nitrite
4. 56.4 grams of chlorine gas	8. 1500 milliliters of 95% ethyl alcohol

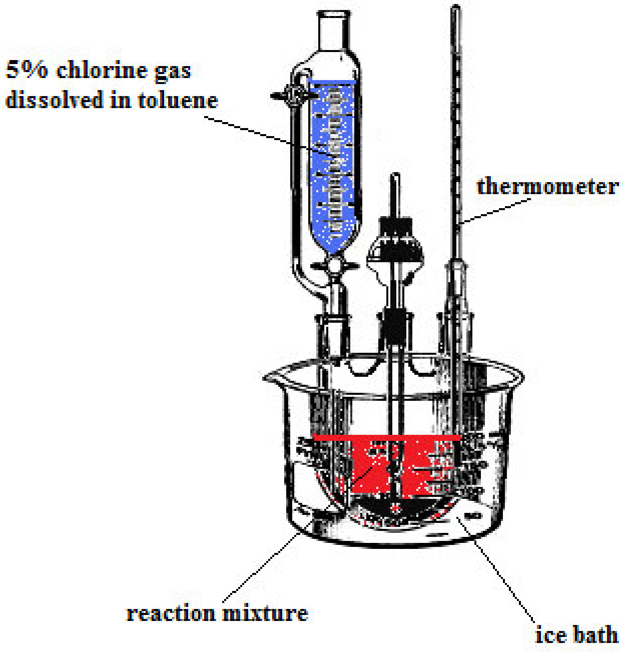
Reaction Equation:


hexa-1,3,5-triyne-1,6-diyl dinitrate
hexane
3Cl2 in Toluene
AlCl3
stir
0 Celsius
evaporate
wash/dry
(1E,3E,5E)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate
6KNO2
Toluene
reflux 120 Celsius
stir
filter 80 Celsius
alcohol
filter
wash/dry
1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate

Preparation:

Step 1: Preparation of (1*E*,3*E*,5*E*)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate

Into an apparatus as similar to the one illustrated below, equipped with motorized stirrer, pour in 500 milliliters of dry hexane, followed by 10 grams of anhydrous aluminum chloride, and then followed by 52 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2). Thereafter, place the apparatus in an ice bath, and chill the reaction mixture to 0 to 5 Celsius. When the temperature of the reaction mixture reaches 0 to 5 Celsius, pour into the addition funnel half of a special solution made by bubbling 56.4 grams of chlorine gas into 1094 grams of toluene (do this action first before proceeding with step 1). Thereafter, turn on the motorized stirrer, and then begin a slow drip of the chlorine/toluene mixture from the addition funnel into the reaction mixture while rapidly stirring the reaction mixture and maintaining its temperature around 0 to 5 Celsius at all times during the addition. Once all the chlorine/toluene mixture has been dripped into the reaction mixture, pour the final half of the pre-prepared mixture of chlorine and toluene into the addition funnel, and continue a slow drip of this chlorine/toluene mixture into the reaction mixture while rapidly stirring the reaction mixture and maintaining its temperature around 0 to 5 Celsius at all times during the addition. After the addition of the second half of the chlorine/toluene mixture to the reaction mixture, continue to rapidly stir the reaction mixture at a temperature around 5 to 0 Celsius for about 4 hours. Note: During this time, the ice bath will have to be periodically changed with fresh ice. Now, after 4 hours, remove the ice bath, and then quickly filter the reaction mixture using suction filtration to remove any insoluble impurities, and then place the filtered reaction mixture into a rotary evaporator, and evaporate-off the hexane and toluene under moderate vacuum and at a temperature of 55 Celsius until dry residue remains. Once dry residue remains, and all the hexane and toluene have been evaporated-off, turn off the rotary evaporator, and collect the left over residue. Finally, place this left over residue into a Buchner funnel fitted with filter paper, and then wash the suction dried residue with six 500-milliliter portions of hot water, and then suction dry as thoroughly as possible, and then save this washed suction dried desired product of (1*E*,3*E*,5*E*)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate for step 2.

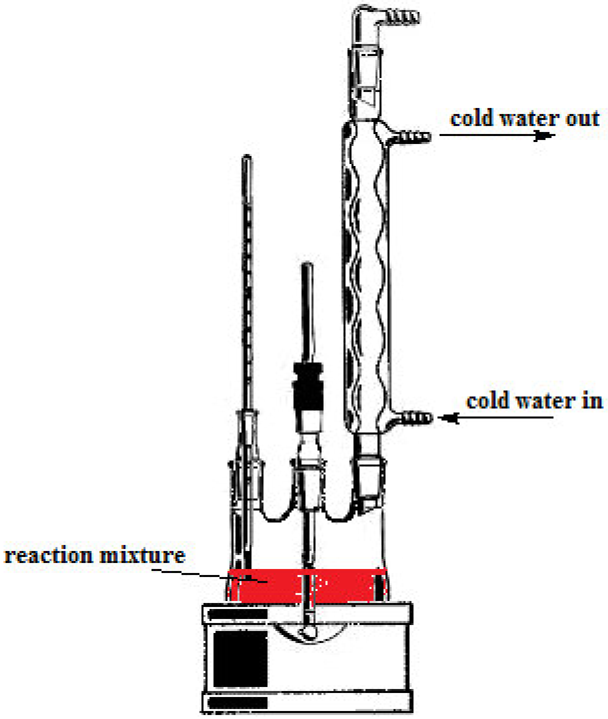


Setup for the chlorination of hexa-1,3,5-triyne-1,6-diyl dinitrate.

Step 2: Preparation of 1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate

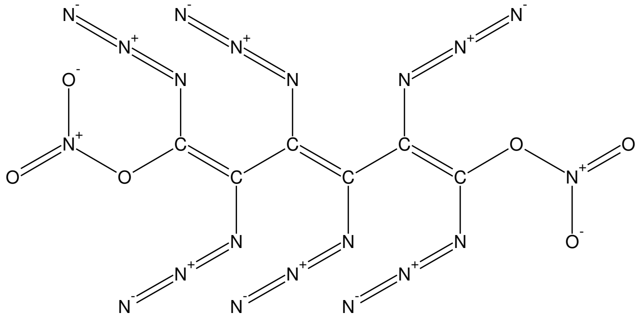
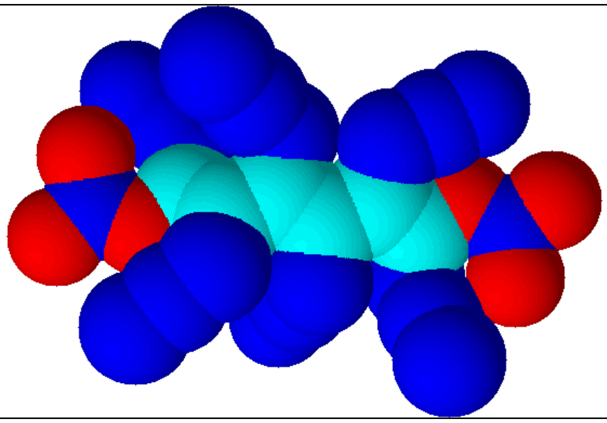
Into a standard reflux apparatus as illustrated below, place 750 milliliters of toluene, followed by the (1*E*,3*E*,5*E*)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate obtained in step 1, and then stir the mixture until the (1*E*,3*E*,5*E*)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate dissolves in the toluene. Once it has, add in 136 grams of potassium nitrite, and then turn on the heating mantle, and bring the reaction mixture to reflux at 120 Celsius with rapid agitation. Then continue to reflux and stir the reaction mixture at 120 Celsius for 6 hours. After 6 hours, turn off the heating mantle, and allow the reaction mixture to cool to about 80 Celsius. When the temperature of the reaction mixture has cooled down to 80 Celsius, quickly disassemble the apparatus, and then suction filter the hot reaction mixture to recover the insoluble potassium chloride by-product. After filtration, pour the filtered hot reaction mixture into a large beaker and allow it to cool to room temperature.

Once it has, pour in 750 milliliters of 95% ethyl alcohol, and then stir the reaction mixture for 2 hours at room temperature. Note: the addition of the alcohol will cause the desired 1,2,3,4,5,6-hexanitrohexa-1,3,5-triene-1,6-diyl dinitrate to precipitate. After stirring the mixture for 2 hours, filter-off the precipitated product using suction filtration, wash with three 250-milliliter portions of fresh 95% ethyl alcohol, and then wash with three 300-milliliter portions of hot water, and suction dry as best as possible. Finally, place the suction dried product into an oven and bake at 105 Celsius for 4 hours. After 4 hours, turn off the oven, and allow the dried desired product to cool to room temperature before removing it for storage.



Setup for nitration of (1E,3E,5E)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate.

Theoretical Preparation 26: (1*Z*,3*E*,5*Z*)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate; EZ-Azido Triene; HAHTDN

 (1*Z*,3*E*,5*Z*)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate	
Expanded structure	3D structure

Molecular data:
Molecular Formula = $C_6N_{20}O_6$
Formula Weight = 448.1946
Composition = C(16.08%) N(62.50%) O(21.42%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available

Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 448.030968 Da
Nominal Mass = 448 Da
Average Mass = 448.1946 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
8009; estimated using infrared spectroscopy	Non-Flammable; Decomposes when rapidly heated to over 400 Celsius, but will not burn.	Very stable. Resistant to shock, heat, percussion, pressure, and friction. A secondary explosive; requires significant blasting cap or detonator for initiation.	Generally non-reactive; does not react with reducing agents, but can deflagrate when heated with strong oxidizers, so use caution.	White, rhombehedric crystals, or white to slightly yellow mass, powder, or granules. Impure compound will have a yellowish tint. Melting point: 127 Celsius, Boiling point: decomposition begins slowly when heated to 280 Celsius, rapid decomposition begin at just over 400 Celsius. Insoluble in water, and most organic solvents.

Estimated Compound summary: (1*Z*,3*E*,5*Z*)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate is an interesting poly azide high explosive that has tremendous stability and power. It has an amazing 62% nitrogen by weight, witch makes it a powerful explosive for use in all the usual explosive munitions. It is readily melted down, and casted into molds, but it cannot be used to make explosive compositions as it is insoluble in most other molten explosives, and has no tendency to set or cure when mixed with plasticizers or epoxies. However, it is readily used by itself either in powder form, dead pressed, or melted down for molds in making a variety of explosive munitions such as shape charges, demolition charges, mortar shells, artillery shells, land mines, grenades, and many others. Due to its high gas ratio of over 80% gas by weight, it most suitable for use in making shape charges for anti-tank weapons; it has tremendous penetrating power and can be used in making double shape charges for use to counter anti-anti-tank reactive armor on tanks. 1 kilogram of this compound will blow a 20mm hole through 14” of solid steel. (1*Z*,3*E*,5*Z*)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate is very water resistance and can be used in manufacturing explosive munitions that will be exposed to the elements for long periods of time. As previously noted, it does however, require a significant blasting cap or detonator for initiation due to its high stability.

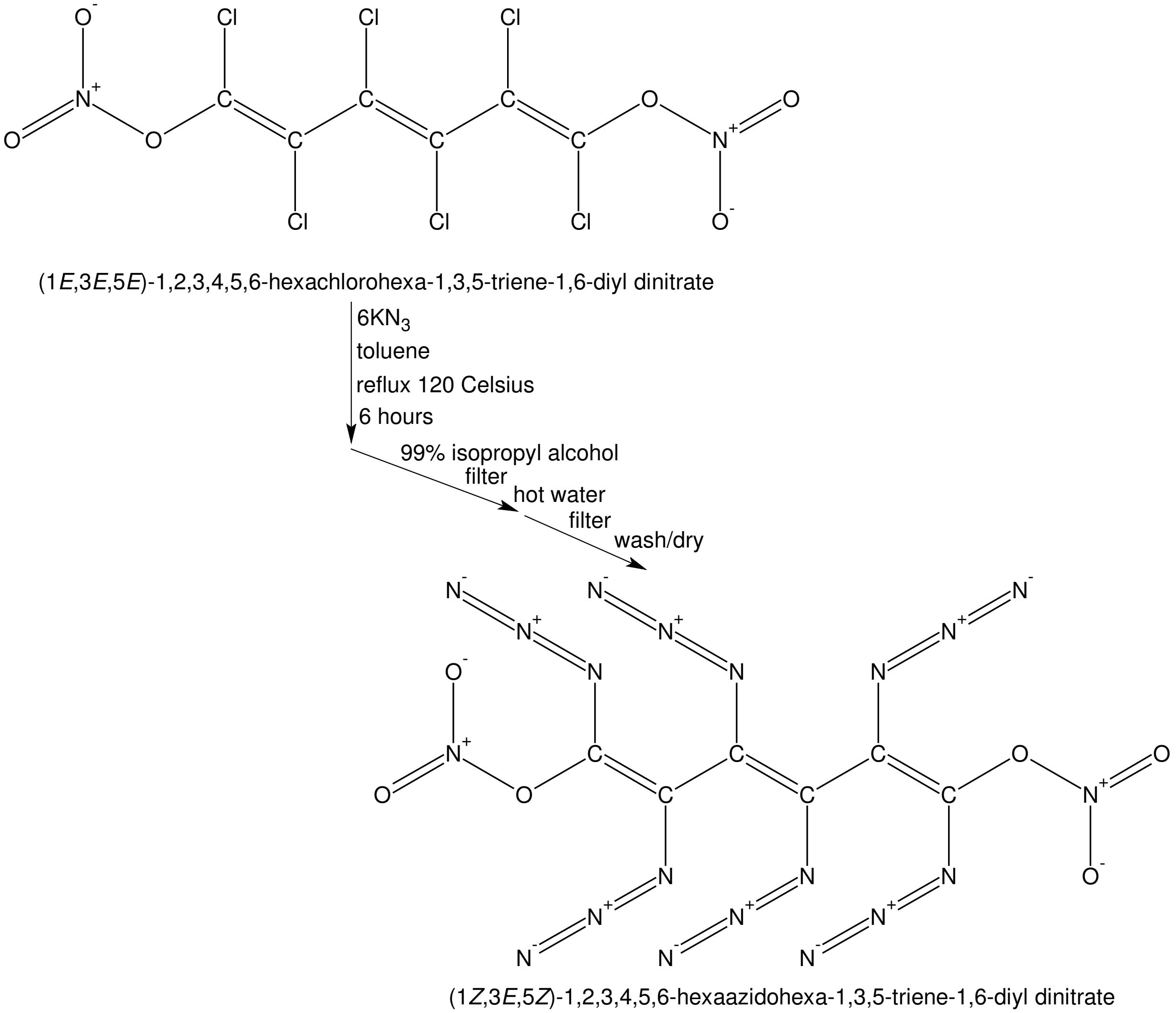
Safety Info: Wear gloves and avoid skin contact and inhalation of toluene, and use proper ventilation when handling as toluene is a known carcinogen. Wear gloves when handling potassium azide, and avoid skin contact, and ingestion as potassium azide is highly toxic.

Materials:

1. 135 grams of (1*E*,3*E*,5*E*)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate (prepared in theoretical	4. 2250 milliliters of 99% isopropyl alcohol

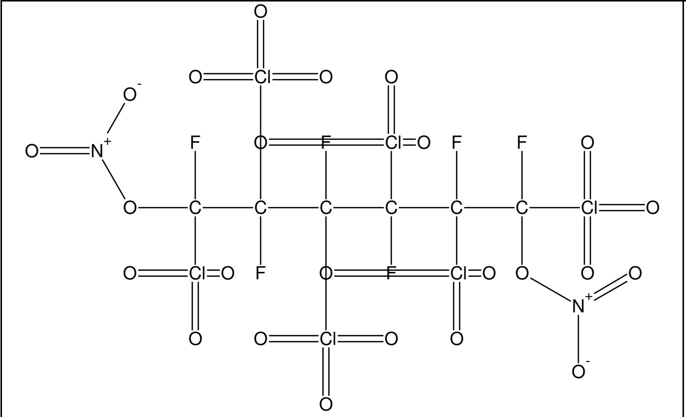
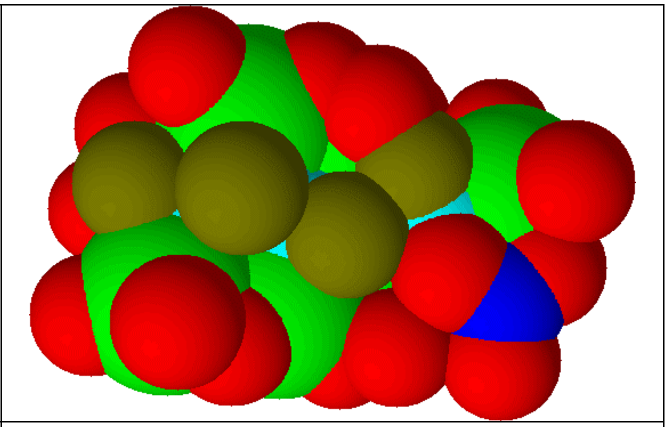
preparation 25, step 1)	
2. 750 milliliters of toluene	3. 161 grams of potassium azide

Reaction Equation:


(1E,3E,5E)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate
6KN3
toluene
reflux 120 Celsius
6 hours
99% isopropyl alcohol
filter
hot water
filter
wash/dry
(1Z,3E,5Z)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate

Preparation: Into a standard reflux apparatus equipped with cold water condenser, thermometer, and motorized stirrer, place 750 milliliters of toluene, and then add in 135 grams of (1*E*,3*E*,5*E*)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate (prepared from theoretical preparation 25, step 1), and then stir the mixture to dissolve the (1*E*,3*E*,5*E*)-1,2,3,4,5,6-hexachlorohexa-1,3,5-triene-1,6-diyl dinitrate. Thereafter, add in 161 grams of potassium azide, and then bring the reaction mixture to reflux by heating to 120 Celsius with constant stirring. Once the reaction mixture begins to reflux at 120 Celsius, continue to rapidly stir the reaction mixture under reflux at 120 Celsius for 6 hours. After 6 hours, remove the heat source and allow the reaction mixture to cool to room temperature. Once it has, disassemble the apparatus, and then pour the reaction mixture into a suitable sized beaker, and then add in 750 milliliters of 99% isopropyl alcohol, and then stir this new reaction mixture for 2 hours at room temperature. After 2 hours, using a large Buchner funnel fitted with proper filter paper, filter-off the insoluble mass using suction filtration, and then suction dry as best as possible. Now, using the same Buchner funnel, wash the suction dried mass in the Buchner funnel three 1500-milliliter portions of hot water to dissolve the potassium chloride by-product. Thereafter, suction dry the desired product of (1*Z*,3*E*,5*Z*)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate as best as possible. Finally, wash the dried suction dried product with three 500-milliliter portions of fresh 99% isopropyl alcohol, and then suction dry as best as possible. Thereafter, place the washed suction dried desired product of (1*Z*,3*E*,5*Z*)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate into an oven, and bake at 100 Celsius for 12 hours. After 12 hours, turn off the oven, and allow the desired product of (1*Z*,3*E*,5*Z*)-1,2,3,4,5,6-hexaazidohexa-1,3,5-triene-1,6-diyl dinitrate to cool to room temperature. Once it has, remove it from the oven, and store in any desired glass or plastic container until use.

Theoretical Preparation 27: 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate; Fluoroperchlorylhexane; HFGPHDD

	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6Cl_6F_6N_2O_{24}$
Formula Weight = 810.7716192
Composition = C(8.89%) Cl(26.24%) F(14.06%) N(3.46%) O(47.36%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 807.687634 Da
Nominal Mass = 808 Da
Average Mass = 810.7716 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7890; estimated by infrared spectroscopy	Non-Flammable; does not burn even under high temperatures	Non-sensitive. Very stable. Non- sensitive to heat, shock, percussion, pressure, and friction. A secondary explosive. Requires blasting cap or detonator for initiation.	Very un-reactive. Does not react with oxidizing agents. Reacts with reducing agents, but only when heated. Slowly decomposes when added to boiling water.	Yellowish solid. Melting point: 89 Celsius. Boiling point: decomposes at 129 Celsius; insoluble in water; from which it slowly hydrolyzes, but slightly soluble in alcohol, acetone, and methylene chloride. Very soluble in hydrofluoric acid, and liquid

				ammonia.

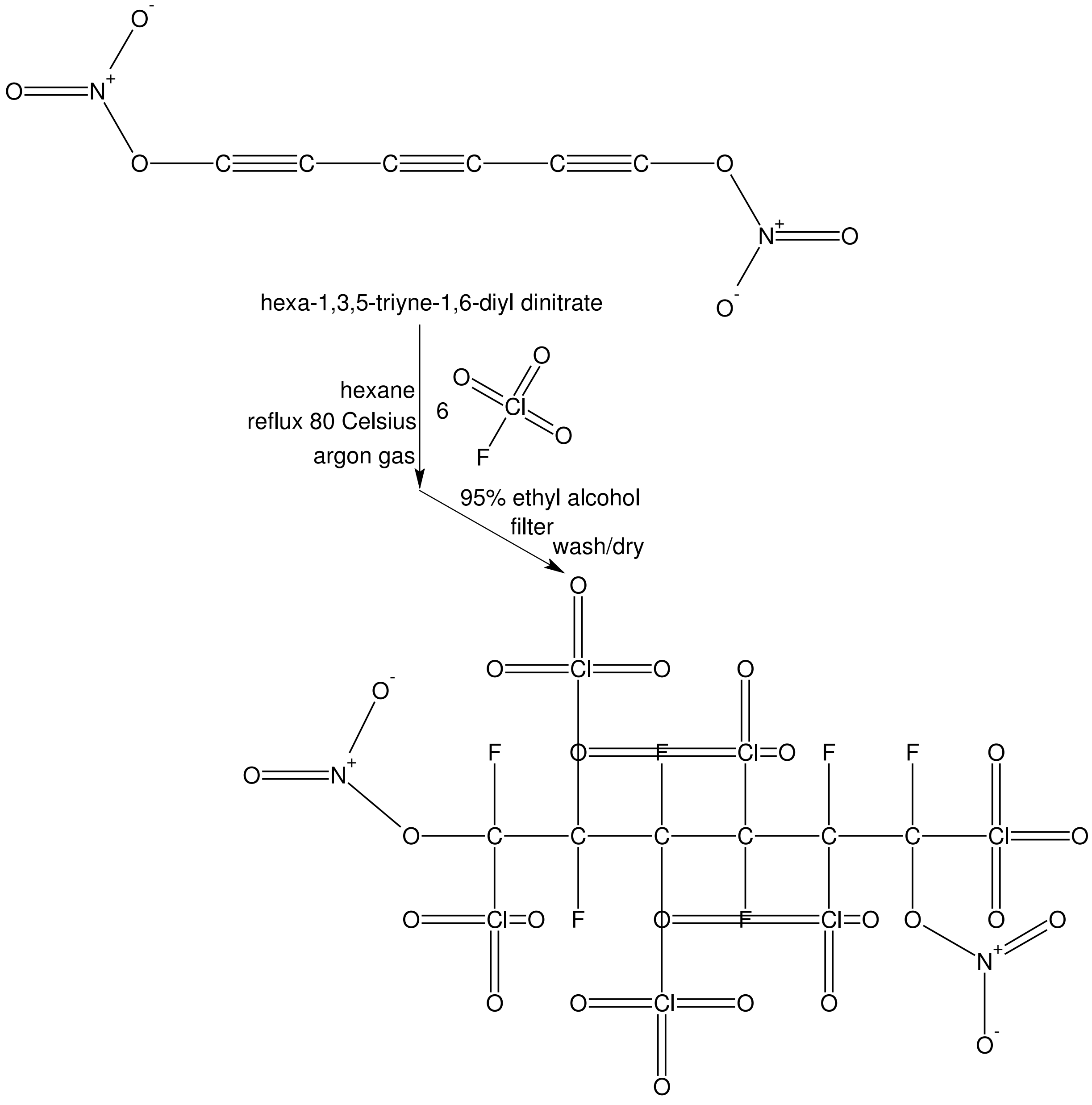
Estimated Compound summary: 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is a high energy plasticizer useful in making explosive compositions when mixed in the molten state with other high explosives to make explosive compositions, for use in making a new type of plastic explosive, which has tremendous power. This plastic explosive can be used in making shape charges, demolition charges, missile warheads, mortar shells, and artillery shells. 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is a high energy plasticizer useful in making high energy high performance rocket propellants, when mixed with reducing agents, and in making high performance gun propellants of the double base type in replacing nitroglycerine and is intended to replace older high energy plasticizers for use in making a variety of explosive high energy munitions. 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is a remarkable high energy plasticizer with great stability, and is almost virtually inert to chemical attack; hence, it can be used in making water resistant and chemical resistant explosive compositions. 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is best used when dead pressed into molds under 2500 psi, or melted down and poured into molds for making a variety of explosive munitions either by-itself or better yet when mixed with other molten explosives such as TNT, RDX, or HMX. It can be used in gelling nitroglycerine, and other poly nitrate glycols for use in nitrate based explosive compositions for many uses. 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate can also be used in gelling gasoline for making explosive/fuel compositions for making hot burning incendiary munitions for burning foliage, buildings, ect., under various conditions. 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is the future of high energy plasticizers with a great deal of chemical resistance with a remarkable explosive power. It can also be used in fireworks, but this type of action has not been thoroughly investigated. 9 grams of 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate equals the explosive power of an M-80 firecracker. In any sense, 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate can be used in making bursting charges for aerial fireworks, but it requires a significant initiator such as a lead azide priming charge, but these charges would be very explosive and most countries would probably ban these sorted fireworks. 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is best used in making plastic explosives when admixed with RDX, TNT, or PETN. 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is by-far one of the best high energy plasticizers, and its use will increase in the future.

Safety Info: Extinguish all flames and sources of ignition before using hexane, which is highly volatile and flammable. Use care when handling perchloryl fluoride, which is very reactive and explosive.

Materials:

1. 750 milliliters of dry hexane	4. 552 grams of perchloryl fluoride gas
2. 176 grams hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2)	5. 2000 milliliters of 95% ethyl alcohol
3. Argon gas from tank	

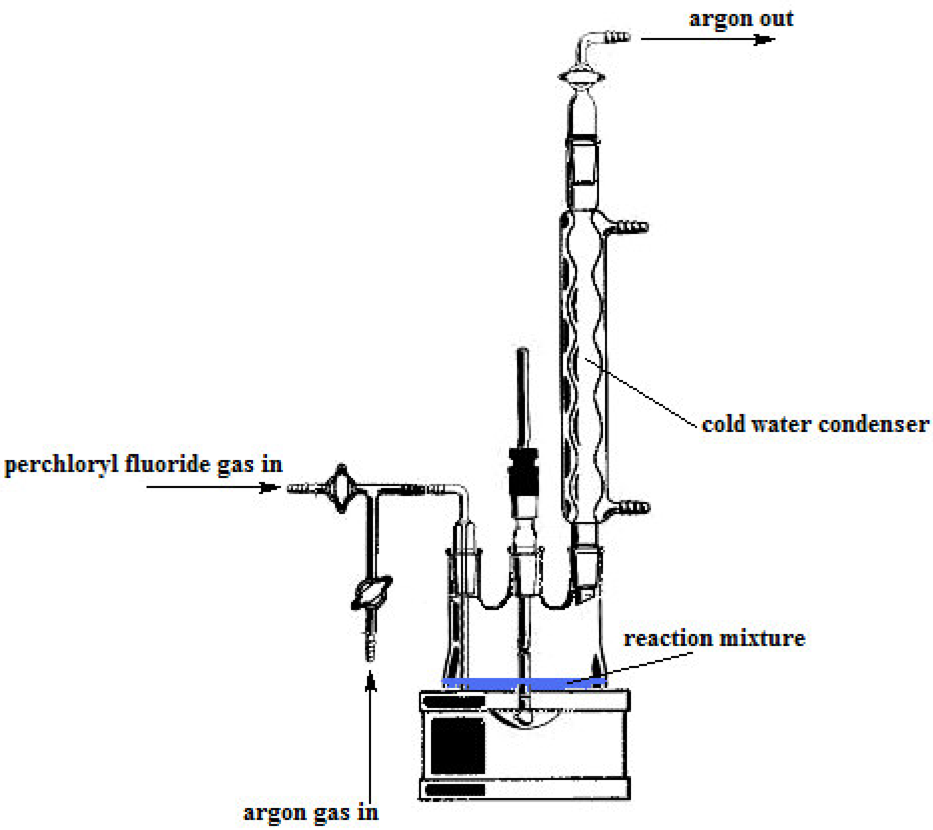
Reaction Equation:



hexa-1,3,5-triyne-1,6-diyl dinitrate

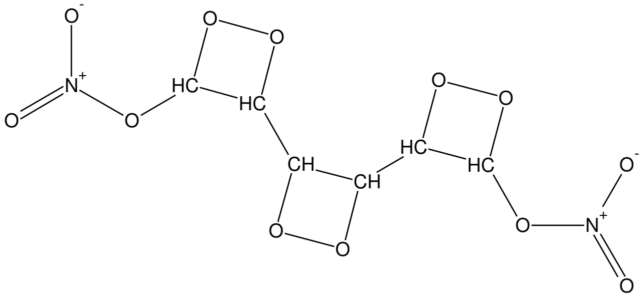
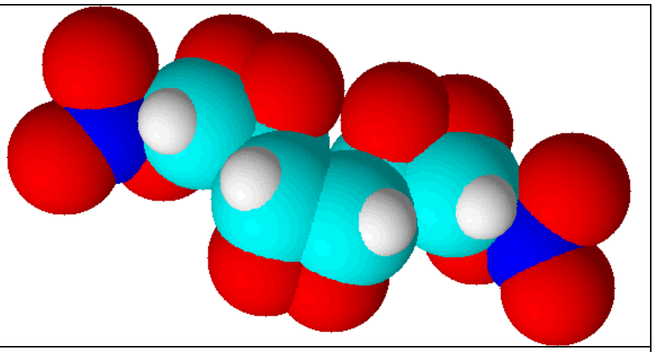
1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate

Preparation: Into an apparatus as illustrated below, place 750 milliliters of dry hexane, and then add in 176 grams hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2), and then stir the mixture for about 10 minutes to form a uniform mixture. Thereafter, begin a argon gas purge by passing into the apparatus dry argon gas at a flow rate of 150 mg per/minute. Thereafter, bring the reaction mixture to a reflux by turning on the heating mantle and heating the reaction mixture to 80 Celsius with constant stirring. When the reaction mixture temperature has reached 80 Celsius, begin to bubble into the reaction mixture dry perchloryl fluoride gas at a flow rate of 50 mg per/minute while continuing to reflux the reaction mixture at 80 Celsius under rapid stirring. Continue to bubble into the reaction mixture dry perchloryl fluoride gas under reflux of 80 Celsius under rapid agitation until 552 grams of perchloryl fluoride gas has been added. Note: continue to purge the apparatus with argon during the entire addition of the perchloryl fluoride. After the addition of the perchloryl fluoride gas, continue to purge the apparatus with argon, and continue to reflux the reaction mixture at 80 Celsius under rapid agitation for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during the cool down period continue to purge the apparatus with argon gas, and continue to the stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus and pour the entire reaction mixture into a large beaker, and then add in 500 milliliters of 95% ethyl alcohol, and then stir this new mixture for 1 hour. After 1 hour, filter-off the insoluble mass using suction filtration, which will be the desired product, and then wash with three 500-milliliter portions of fresh 95% ethyl alcohol, followed by three 500-milliliter portions of hot water, and then suction dry as best as possible. Finally place the suction dried product onto a shallow pan, and allow it to air-dry for 48 hours; after which the desired product of 1,2,3,4,5,6-hexafluoro-1,2,3,4,5,6-hexaperchlorylhexane-1,6-diyl dinitrate is ready for use.



Setup for addition of perchloryl fluoride to hexa-1,3,5-triyne-1,6-diyl dinitrate.

Theoretical Preparation 28: 3,3':4',3''-ter-1,2-dioxetane-4,4''-diyl dinitrate; Dioxetane; Dioxetane dinitrate; ter-DDD

 3,3':4',3''-ter-1,2-dioxetane-4,4''-diyl dinitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6H_6N_2O_{12}$
Formula Weight = 298.11804
Composition = C(24.17%) H(2.03%) N(9.40%) O(64.40%)
Molar Refractivity = 49.57 ± 0.4 cm^3
Molar Volume = 150.0 ± 5.0 cm^3

Parachor = 449.9 ± 6.0 cm^3
Index of Refraction = 1.574 ± 0.03
Surface Tension = 80.8 ± 5.0 dyne/cm
Density = 1.98 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 19.65 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 297.992074 Da
Nominal Mass = 298 Da
Average Mass = 298.118 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
5980	Non-flammable. Will not burn unless heated to over 350 Celsius, from which it burns with a reddish flame emitting toxic fumes of nitrogen oxides and carbon monoxide. Burns violently when mixed with reducing agents and ignited.	Stable at ordinary temperatures. Non-sensitive to pressure, shock, friction, and percussion; however when struck with a heavy hammer, a small sample will detonate, and a small sample will detonate when struck by a rifle bullet. A secondary explosive. Requires a blasting cap or detonator for initiation.	Generally reactive; reacts with strong bases forming shock sensitive salts; reacts with halogens, and explodes in contact with fluorine.	Colorless monoclinic crystals, white powder or granules. Melting point: 190 Celsius; Boiling point: Explodes when heated to 500 Celsius. The compound is insoluble in water, and most organic solvents, but has slight solubility in acetone, alcohol, and ether.

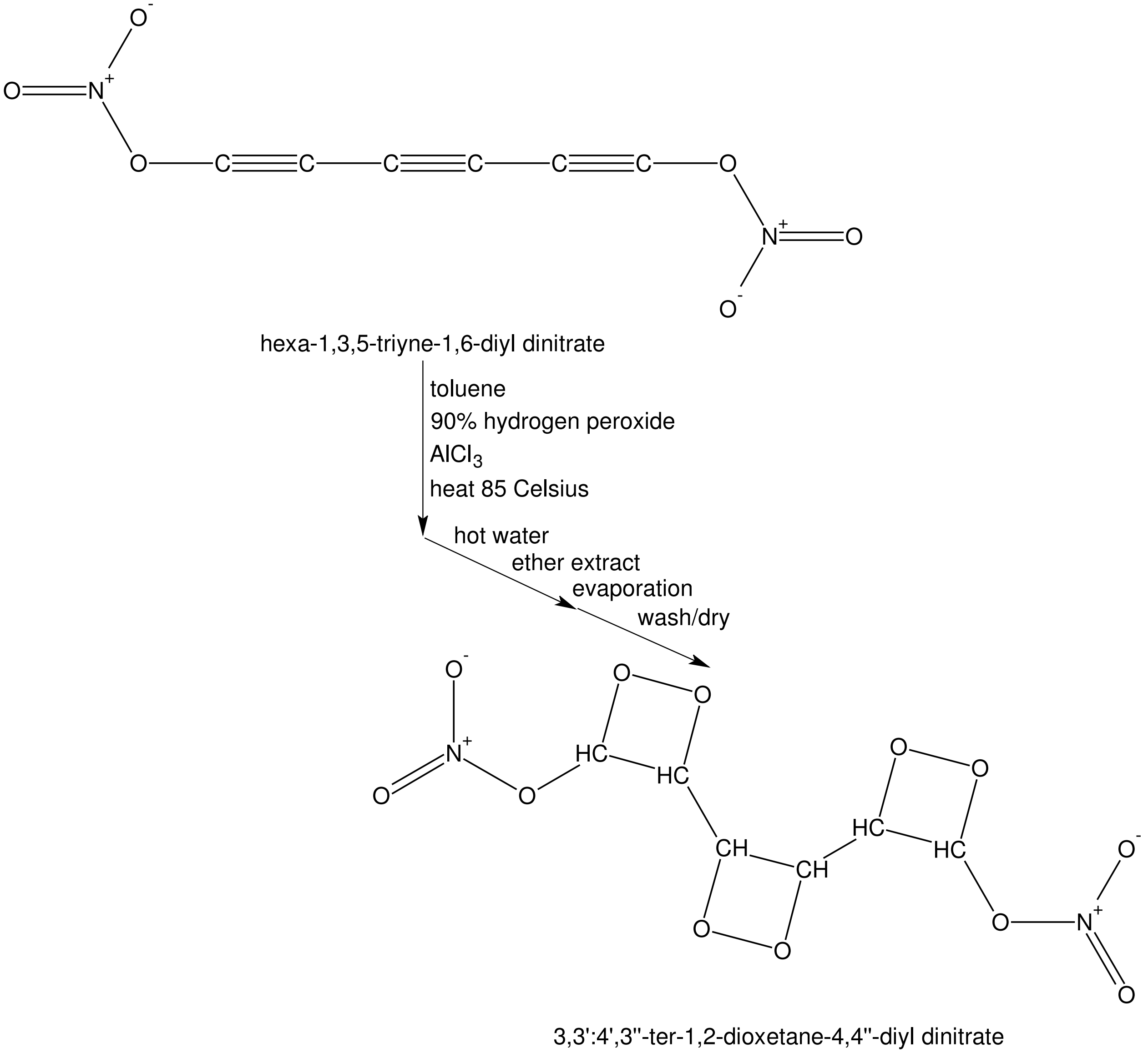
Estimated Compound summary: 3,3':4',3"-ter-1,2-dioxetane-4,4"-diyl dinitrate is a new peroxide based nitrate explosive that is primarily used to make primary explosives as it reacts with strong bases and powdered aluminum or zinc dust forming explosives that are easily detonated. 3,3':4',3"-ter-1,2-dioxetane-4,4"-diyl dinitrate is a interesting compound that warrants further investigation. It can be used in many types of munitions, but because of its rather low detonating velocity, it's use is primarily strictly used in making demolition charges when mixed with ammonium nitrate for blasting soft and hard rock for construction and mining purposes. 3,3':4',3"-ter-1,2-dioxetane-4,4"-diyl dinitrate has a high melting point making it suitable for use in explosive munitions that will be exposed to hot weather conditions, but it cannot be melted down and mixed with other molten explosives due to its lack of solubility; however, it can be melted down by itself and poured into molds for use in making shape charges, mortar rounds, artillery shells, and other explosive munitions, but it should not be used in high velocity projectiles such as tank fired projectiles due to its tendency to explode prematurely.

Safety Info: Wear gloves and use proper ventilation when handling toluene, which is known carcinogen, avoid skin contact and inhalation. Wear gloves when handling anhydrous aluminum chloride, and avoid contact with water as it reacts violently with water. Use caution when handling 90% hydrogen peroxide and avoid contact with reducing agents as violent reactions may take place. Use proper ventilation when handling diethyl ether and extinguish all sources of ignition as diethyl ether is highly volatile and forms explosive mixtures with air. Also, never use old diethyl ether as it may contain peroxides, which are explosive in nature.

Materials:

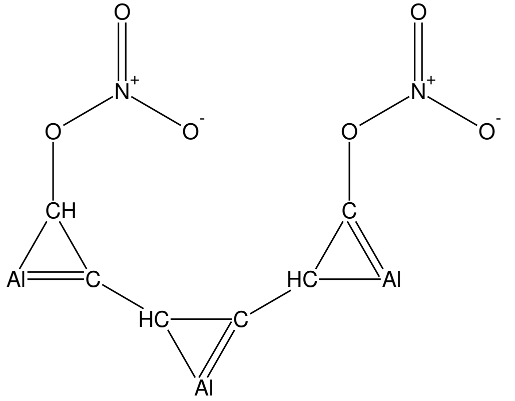
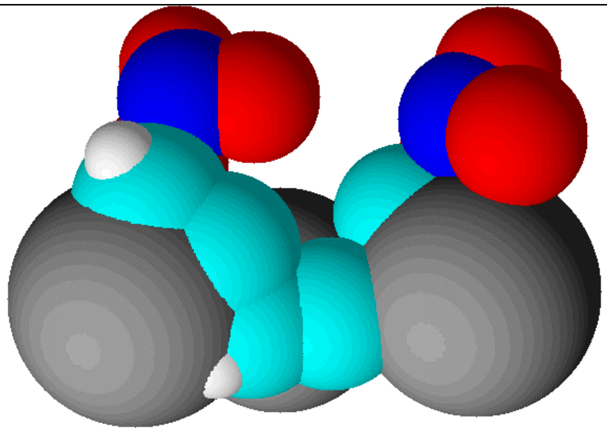
1. 750 milliliters of dry toluene	4. 80 grams of 90% hydrogen peroxide
2. 137 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2)	5. 2500 milliliters of dry diethyl ether
3. 25 grams of anhydrous aluminum chloride	

Reaction Equation:



Preparation: Into a suitable 3-neck flask in a heating mantle and equipped with motorized stirrer, condenser, and addition funnel, place 750 milliliters of dry toluene, and then add in 137 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2), followed by 25 grams of anhydrous aluminum chloride, and then stir the mixture for 15 minutes to form a uniform mixture. Thereafter, pour into the addition funnel 80 grams of 90% hydrogen peroxide, and then turn on the heating mantle and heat the contents in the 3-neck flask to 85 Celsius with constant stirring. When the contents in the 3-neck flask reaches 85 Celsius, begin a slow drip of the 90% hydrogen peroxide from the addition funnel into the reaction mixture over a period of about 45 to 50 minutes while heating the reaction mixture to 85 Celsius under rapid stirring. After the all the 90% hydrogen peroxide has been added, continue to heat the reaction mixture at 85 Celsius under vigorous stirring for 6 hours. After 6 hours, turn off the heating mantle and allow the reaction mixture to cool to room temperature. Note: continue to vigorously stir the reaction mixture during this cool down period. When the reaction mixtures temperature reaches room temperature, disassemble the apparatus, and then filter-off the insoluble material that will contain the desired product mixed with the anhydrous aluminum chloride using a Buchner funnel under suction filtration. Now, to remove the aluminum chloride wash the suction filtered material with three 150-milliliter portions of hot water, and then suction dry as best as possible. Now, to the washed suction dried material remaining in the Buchner funnel, remove it, and place it into a large beaker, and then pour in 2500 milliliters of dry diethyl ether and then stir this mixture to dissolve the desired product into the ether. Thereafter, filter this ether mixture to remove insoluble impurities, and then place the filtered ether mixture into a rotary evaporator and evaporate-off the ether under mild vacuum at a temperature of 35 Celsius until dry solid remains. Once all the ether has been removed, and dry solid remains, stop the evaporation process, and then recover the left over dried residue, which will be the purified desired product. Finally, place this dried left over product residue into a clean Buchner funnel, and then wash it with three 500-milliliter portions of warm water, and then suction dry as best as possible. Then place this suction dried product of 3,3':4',3"-ter-1,2-dioxetane-4,4"-diyl dinitrate into an oven, and bake at 105 Celsius for 12 hours. After 12 hours, turn off the oven, and allow the desired product of 3,3':4',3"-ter-1,2-dioxetane-4,4"-diyl dinitrate to cool to room temperature before removing it. After which, it can be stored in any desired container until use.

Theoretical Preparation 29: 2*H*,3'*H*,3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate; Aluminum-3H-dinitrate; Aluminum-3-3-dinitrate; 2H’3H’-Aluminum dinitrate

 O N+ O O- CH Al C HC C Al O N+ O O- C HC Al 2*H*,3'*H*,3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6H_3Al_3N_2O_6$

Formula Weight = 280.0424358

Composition = C(25.73%) H(1.08%) Al(28.90%) N(10.00%) O(34.28%)

Molar Refractivity = Not available

Molar Volume = Not available

Parachor = Not available

Index of Refraction = Not available

Surface Tension = Not available

Density = Not available

Dielectric Constant = Not available

Polarizability = Not available

Monoisotopic Mass = 279.943725 Da

Nominal Mass = 280 Da

Average Mass = 280.0424 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
4579; estimated using infrared spectroscopy	Spontaneously combustible in air. Explodes when heated.	Sensitive to shock, percussion, and friction. Easily detonated by spark or flame. A primary explosive.	Very reactive. Reacts violently with water, spontaneously ignites in air, reacts with the halogens with generation of great heat; explodes in	Yellowish crystalline needle like crystals. Melting point: explodes; boiling point: N/A. Insoluble in water

			contact with fluorine. Reacts with alky and aryl halides.	from which it reacts violently as previously stated; insoluble in most organic solvents (reacts with many solvents and may ignite spontaneously with ether), but soluble in liquid ammonia.

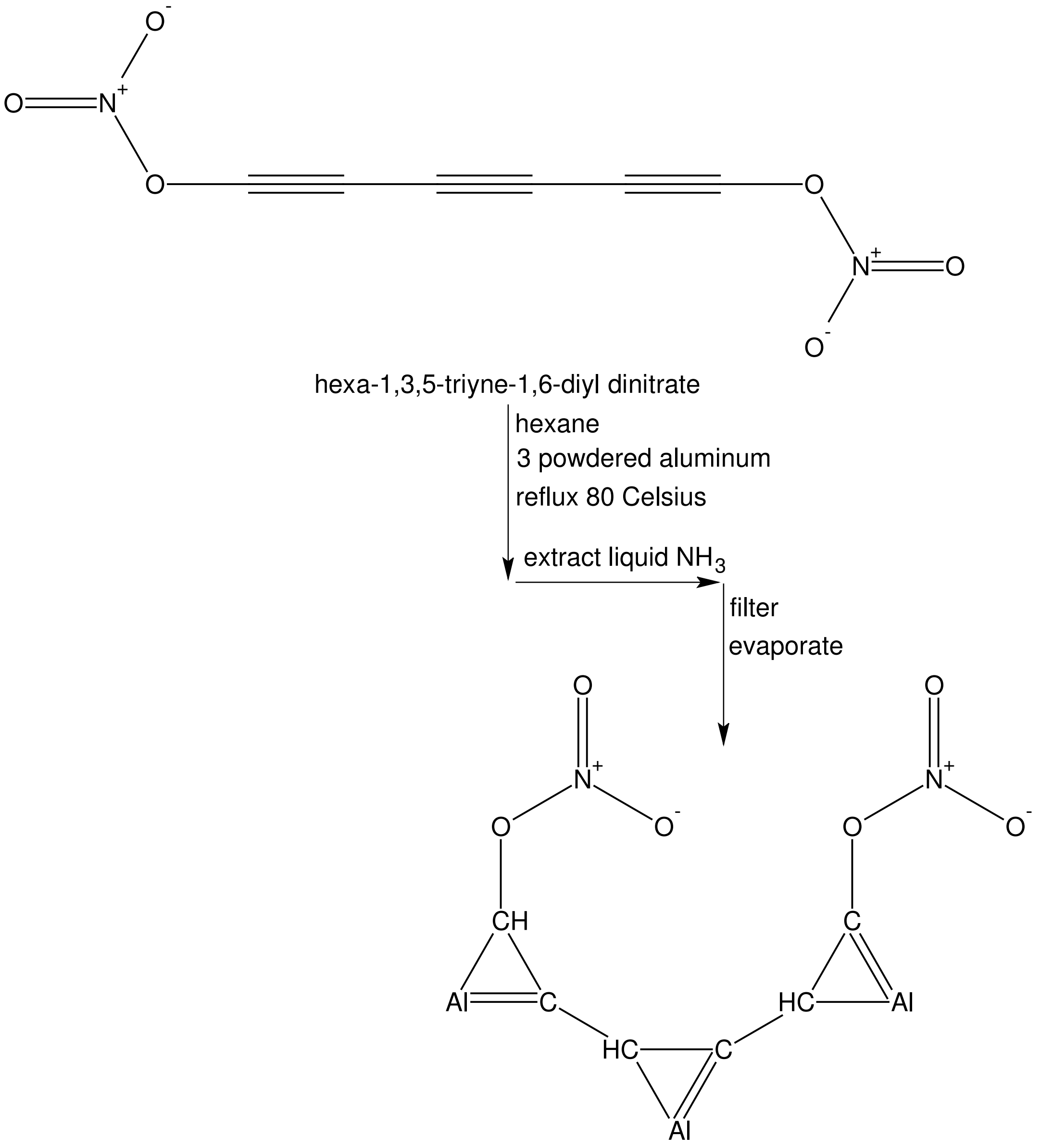
Estimated Compound summary: 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate is a radical aluminum based primary explosive. Although it ignites spontaneously in air, it is an excellent primary explosive for use in blasting caps and detonators. Note: these blasting caps and detonators must be sealed water and air tight. 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate is easily detonated by spark or flame, and hence makes a perfect primary explosive to replace the aging lead styphnate/barium chromate ignition charge, or for replacing the aging lead azide booster charge in blasting caps and detonators. Even though 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate is sensitive to shock, friction, and percussion, it is however more stable then primary explosives like mercury fulminate and lead azide. The only draw back to 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate is its tendency to spontaneously ignite in air, and this can pose a problem during the manufacturing of the compound and its loading into blasting caps and detonators; nevertheless, it's use as a primary explosive is perfect for priming all kinds of secondary explosives. 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate can also be pressed into tubing for use in making easy to ignite detonating cord; however, this is highly unlikely to replace the common PETN in detonator cord. 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate is one of many aluminum containing diyl dinitrates, and is one of the best, even though its major drawback is its reactivity. In one interesting note, instead of using 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate as an explosive, it can be pressed into shells, grenades, and other low velocity munitions for use in incendiary purposes; however it should not be used in high velocity projectiles due to its sensitivity and tendency to detonate prematurely as excepted. It's best use as an incendiary agent is in the form of hand thrown grenades, or low pressure low velocity weapons such as a 40mm grenade launchers, and fitting these devises with small TNT bursting charges to spread the agent out over a significant area to be burned.

Safety Info: Extinguish all flames and sources of ignition when handling hexane, which is highly volatile and flammable. Use care when handling powdered aluminum and keep it away from any source of ignition, as powdered aluminum is highly flammable. Use care and proper ventilation when handling liquid ammonia, which is highly volatile and emits nauseous fumes.

Materials:

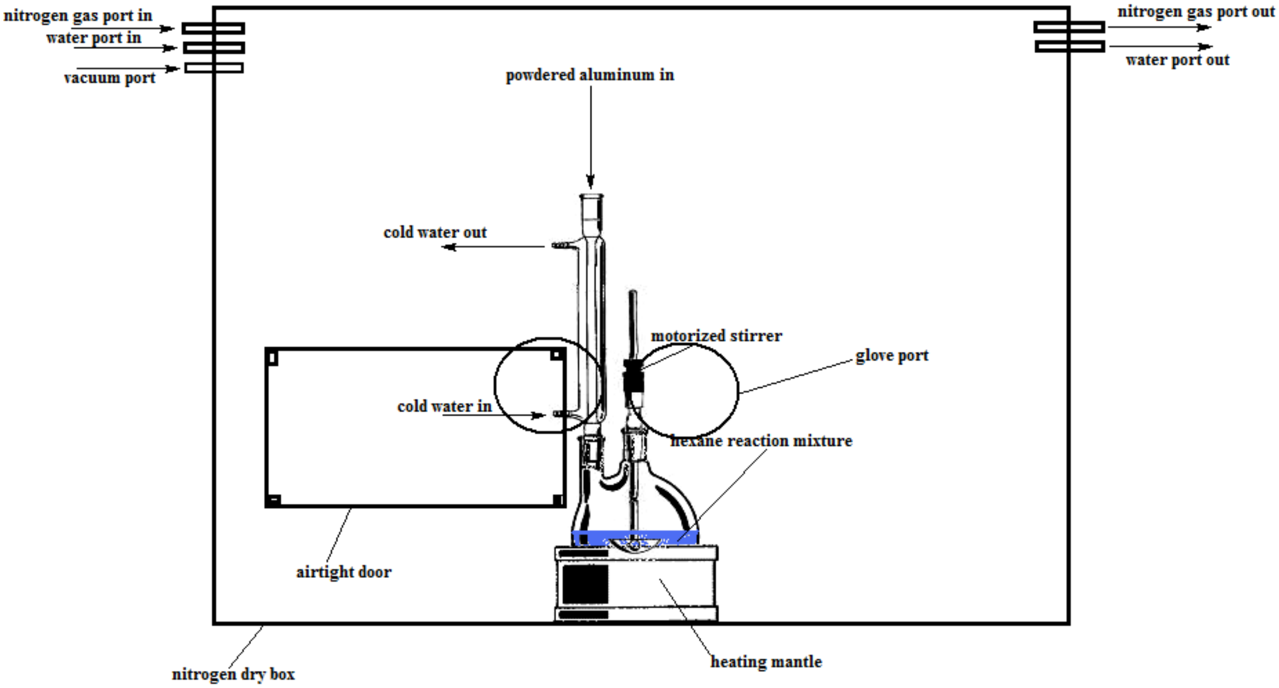
1. 750 milliliters of dry hexane	3. 62 grams of powdered aluminum
2. 149 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2)	4. 1200 milliliters of liquid ammonia

Reaction Equation:



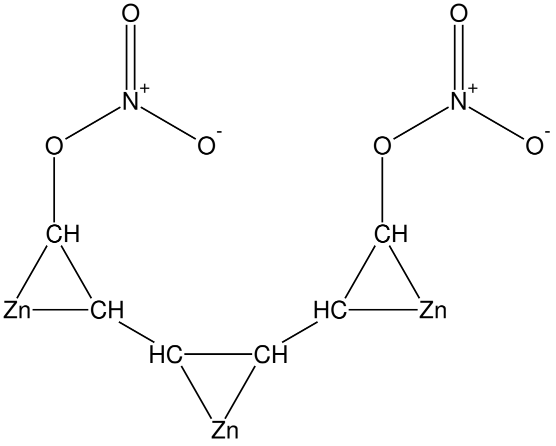
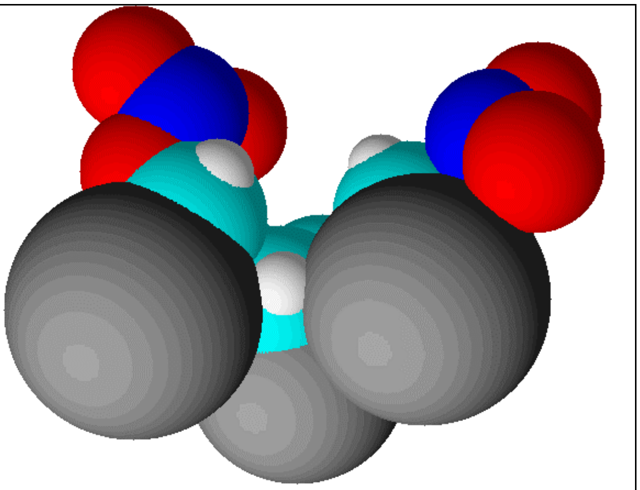
2*H*,3'*H,*3''*H*-2,2':3',2''-teraluminum-3,3''-diyl dinitrate

Preparation: Inside a nitrogen dry box, free from air/oxygen, assemble an apparatus by putting a 2-neck flask into a heating mantle equipped with motorized stirrer, and cold-water condenser. Note: this nitrogen dry box is crudely illustrated but is a small box about 3 feet wide by 3 feet high, with hose connections for purging the inside of the box with an inert atmosphere and hose connections for water, and has exit ports for venting injected gas and an exit port for the exit of water, and has a single port door that opens and closes for putting in lab equipment, and this door seals air-tight. The nitrogen dry box also has two airtight sealed glove holes for putting hands into thereby allowing the user to handle lab equipment. Now, open the nitrogen dry box, and pour into the 2-neck flask 750 milliliters of dry hexane, followed by 149 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2), and then stir the mixture for 15 minutes to form a uniform mixture. Thereafter, place 62 grams of powdered aluminum onto a watch glass and open the door to the nitrogen dry box and place this watch glass inside the nitrogen dry box, and then close the door to the nitrogen dry box, and then purge the nitrogen dry box with pure nitrogen to flush out any air. Now, turn on the heating mantle, and heat the hexane to 80 Celsius under reflux conditions. When the temperature of the contents in the 2-neck flask reaches 80 Celsius, add in, through the open top of the cold-water condenser 62 grams of powdered aluminum from the watch glass in small portions at a time (500 mg per portion) while refluxing the reaction mixture and stirring it vigorously during the addition. After the addition of the powdered aluminum, which should take about 50 minutes, continue to reflux the reaction mixture at 80 Celsius with rapid stirring for 4 hours. After 4 hours, turn off the heating mantle and allow the reaction mixture to cool to room temperature, but continue to stir the reaction mixture until the reaction mixture has cooled. Thereafter, turn off the stirrer and allow the reaction mixture to settle (the desired product will settle to the bottom of the 2-neck flask under the hexane thereby protecting it from air and moisture. Now, disassemble the apparatus and then open the airtight door to the nitrogen dry box, and quickly insert a Buchner funnel with flask, and then close the airtight door, and continue to purge the nitrogen dry box with nitrogen gas to exclude oxygen. Thereafter, filter-off the insoluble product material from the 2-neck flask using suction filtration, and then suction dry as best as possible. Thereafter, re-open the airtight door to the nitrogen dry box, and insert a clean Buchner funnel and flask followed by a beaker containing 1200 milliliters of liquid ammonia, and then seal the nitrogen dry box and continue to purge it to remove oxygen. Now, take the suction dried product from the Buchner funnel and add it to the beaker containing the liquid ammonia, and then stir this mixture vigorously for 30 minutes to dissolve the desired product into the liquid ammonia. After 30 minutes, using the clean Buckner funnel, filter the liquid ammonia mixture to remove insoluble impurities, and then allow the filtered liquid ammonia to evaporate until dry solid remains. Note: allow the liquid ammonia to evaporate under its own force while continuing to purge the nitrogen dry box with nitrogen; the gaseous ammonia will simply be carried away along with the nitrogen out through the out port. Once dry solid remains, the desired product of 2*H*,3'*H,*3"*H*-2,2':3',2"-teraluminum-3,3"-diyl dinitrate can be collected and is ready for use. Note: this desired product should be stored under hexane until use.



Nitrogen dry box setup for preparing 2H,3'H,3"H-2,2':3',2"-teraluminum-3,3"-diyl dinitrate.

Theoretical Preparation 30: 2,2':3',2''-terzincane-3,3''-diyl dinitrate; Terizincane; TZDD

 2,2':3',2''-terzincane-3,3''-diyl dinitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6H_6N_2O_6Zn_3$
Formula Weight = 398.34864
Composition = C(18.09%) H(1.52%) N(7.03%) O(24.10%) Zn(49.26%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 393.810024 Da
Nominal Mass = 394 Da
Average Mass = 398.3486 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
4900; estimated using infrared spectroscopy	Flammable solid in air. Will not burn in absence of oxygen. In presence of oxygen burns forming nitrogen oxides, water, zinc oxide, and carbon monoxide.	Moderately sensitive to shock, percussion, but stable towards friction, heat, and pressure. A sample of compound will detonate when struck by a hammer, or when touched by a spark or flame. A Primary explosive.	Hydrolyzes by moisture and water. Reacts vigorously with the halogens; detonates in contact with fluorine. It is oxidized by strong oxidizers, and may ignite in contact with potassium permanganate.	White crystalline solid, powder, or granules. Melting point: 97 Celsius when slowly heated, but detonates when rapidly heated, Boiling point: decomposes. Insoluble in water from which it is hydrolyzed and

				most organic solvents. Soluble in liquid sulfur dioxide and liquid ammonia.

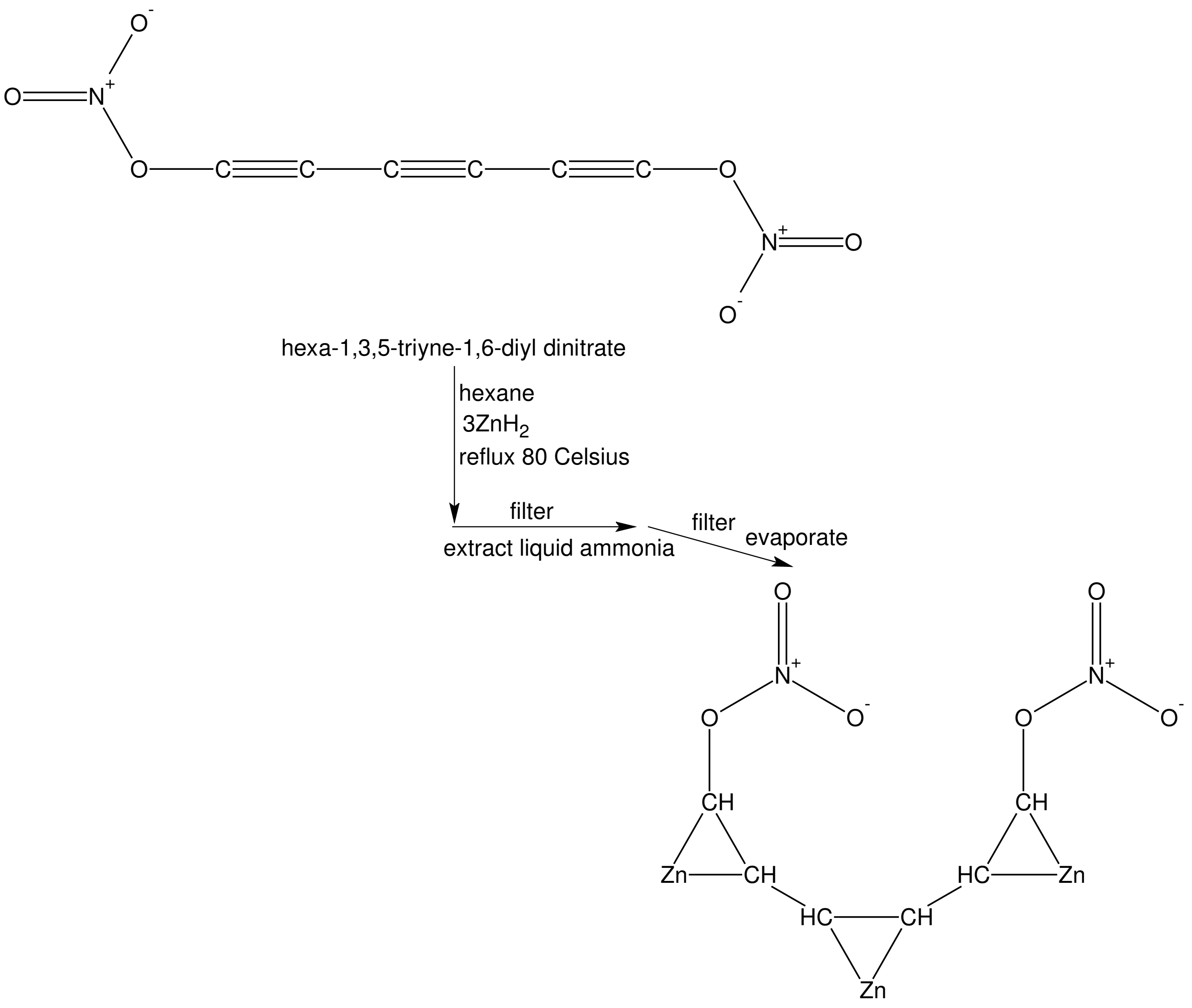
Estimated Compound summary: 2,2':3',2"-terzincane-3,3"-diyl dinitrate is a zinc containing primary explosive, that is well suitable for use in making primers as it detonates when struck by a firing pin or anvil, and is well suitable for replacing lead styphnate or lead azide for use in blasting caps or detonators. 2,2':3',2"-terzincane-3,3"-diyl dinitrate can also be used in making priming cord in place of PETN. The compound is well suitable for priming many types of secondary explosives. 2,2':3',2"-terzincane-3,3"-diyl dinitrate is much safer to handle and manufacture then mercury fulminate, and is more stable then lead azide, as it does not need to be diluted with fillers for stability. Due to its lack of water resistance it must be dead pressed into watertight blasting caps and detonators, but even then it should not be used in explosive munitions that will be exposed to the elements for prolonged periods of time. 2,2':3',2"-terzincane-3,3"-diyl dinitrate unlike its aluminum counter part can be used in high velocity projectiles for priming main secondary charges. It can also be used in mortar shells, and artillery shells, but should not be used in land mines due its lack of water resistance. 2,2':3',2"-terzincane-3,3"-diyl dinitrate is relatively stable when kept dry, and can be stored for years without any worries of decomposition or explosion. 2,2':3',2"-terzincane-3,3"-diyl dinitrate also shows remarkable future use as an ingredient in high performance gun propellants for making double based gun powders to replace the aging nitroglycerine and nitrocellulose for use in small arms, artillery propellants and for igniting propellant charges in mortar rounds. 2,2':3',2"-terzincane-3,3"-diyl dinitrate also shows some potential use in making bursting charges for aerial fireworks. 2,2':3',2"-terzincane-3,3"-diyl dinitrate detonates when rapidly heated, but if gently and slowly heated up to 97 Celsius, it melts forming a colorless liquid, which can be molded into many shapes for various uses, but should not be used to make shape charges or demolition charges due to its ease of detonation. Note: molten 2,2':3',2"-terzincane-3,3"-diyl dinitrate is insoluble in most molten secondary explosives.

Safety Info: Extinguish all flames and sources of ignition when handling hexane, which is highly volatile and flammable. Use care when handling powdered zinc hydride, which reacts violently with water. Use care and proper ventilation when handling liquid ammonia, which is highly volatile and emits nauseous fumes.

Materials:

1. 750 milliliters of dry hexane	3. 200.5 grams of powdered zinc hydride
2. 207 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2)	4. 1500 milliliters of liquid ammonia

Reaction Equation:

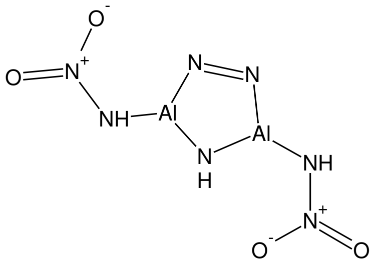
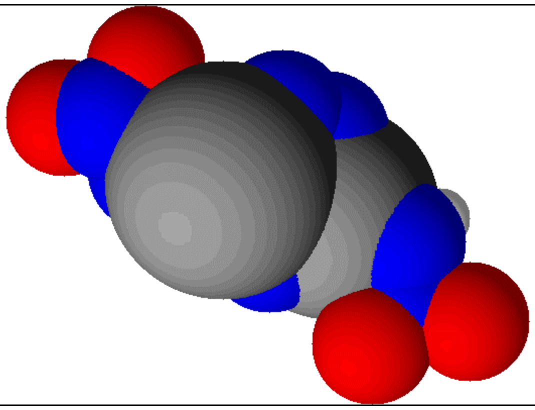


hexa-1,3,5-triyne-1,6-diyl dinitrate

2,2':3',2"-terzincane-3,3"-diyl dinitrate

Preparation: Into an apparatus using a 3-neck flask equipped with heating mantle, motorized stirrer, condenser, and thermometer, pour into the 3-neck flask 750 milliliters of dry hexane, followed by 207 grams of hexa-1,3,5-triyne-1,6-diyl dinitrate (prepared in theoretical preparation 24, step 2), and then stir the mixture for 15 minutes to form a uniform mixture. Thereafter, turn on the heating mantle, and heat the hexane to 80 Celsius under reflux conditions. When the temperature of the contents in the 3-neck flask reaches 80 Celsius, add in, through the open top of the cold-water condenser 200.5 grams of powdered zinc hydride in small portions at a time (500 mg per portion) while refluxing the reaction mixture and stirring it vigorously during the addition. After the addition of the powdered zinc hydride, which should take about 6 hours, continue to reflux the reaction mixture at 80 Celsius with rapid stirring for 2 hours. After 2 hours, turn off the heating mantle and allow the reaction mixture to cool to room temperature, but continue to stir the reaction mixture until the reaction mixture has cooled. Thereafter, turn off the stirrer and allow the reaction mixture to settle (the desired product will settle to the bottom of the 3-neck flask under the hexane. Now, disassemble the apparatus and filter-off the insoluble product material from the 3-neck flask using a Bucher funnel under suction filtration, and then suction dry as best as possible. Thereafter, take the filtered off suction dried material from the Buchner funnel, and add it to a beaker containing 1500 milliliters of liquid ammonia, and then vigorously stir this mixture for 30 minutes to dissolve the desired product into the liquid ammonia. After 30 minutes, filter the liquid ammonia mixture using a clean Buchner funnel of proper size under suction to remove insoluble impurities, and thereafter allow the filtered liquid ammonia to evaporate until dry solid remains. Note: allow the liquid ammonia to evaporate under its own force, but use proper ventilation. Once dry solid remains, the desired product of 2,2':3',2"-terzincane-3,3"-diyl dinitrate can be collected and is ready for use. Note: this desired product should be stored in a dessicator over concentrated sulfuric acid until use.

Theoretical Preparation 31: 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium; Aluminatriazole dinitramine; bis-NADAT; XL-90

 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_3Al_2N_7O_4$
Formula Weight = 219.0313972
Composition = H(1.38%) Al(24.64%) N(44.76%) O(29.22%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 218.987728 Da
Nominal Mass = 219 Da
Average Mass = 219.0314 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6987; estimated	Non-flammable at ordinary temperatures, but will burn in excess oxygen when heated above 200 Celsius.	Stable. Non-sensitive to shock, percussion, heat, pressure, and friction. Drop test: 1 Kg weight = 9 meters. A secondary explosive. Requires a blasting cap or detonator for initiation.	Generally non-reactive. Slowly hydrolyzes in water, and is oxidized by strong oxidizing agents. Explodes in contact with fluorine. May ignite if mixed with potassium permanganate and heated or in contact with flame. Reacts with strong bases, and alkali metals producing shock	Yellowish, needle-like crystals, powder, or granules. Melting point: 105 Celsius; boiling point: decomposes when heated to 230 Celsius. Insoluble in water and slowly hydrolyzed by it. Insoluble in most organic solvents. Very slightly soluble in acetone

			sensitive salts that easily detonate.	and ether. Insoluble in liquid ammonia.

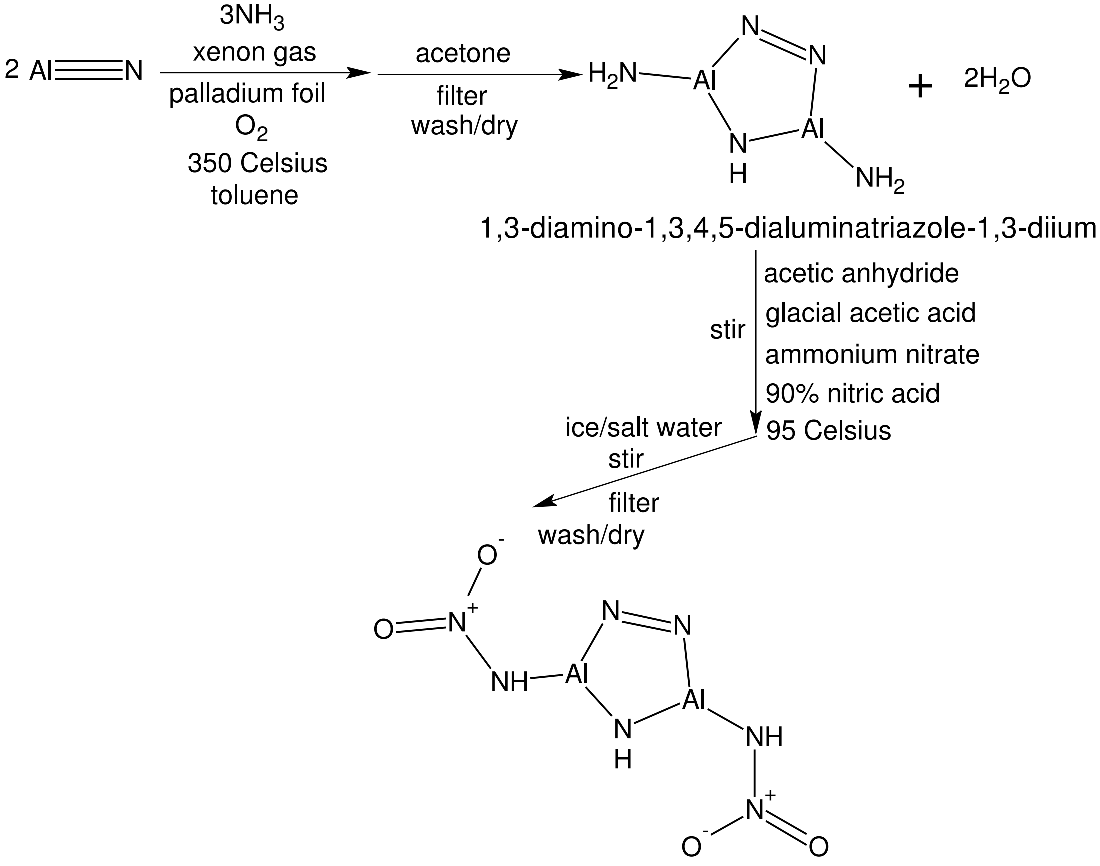
Estimated Compound summary: 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium is a rather interesting aluminum containing triazole explosive with two nitramine groups attached making it a powerful explosive. The stability of this compound is rather unique and surprising, but it does slowly hydrolyze by water; nevertheless, 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium shows remarkable stability and with its rather low melting point it can be melted down and poured into molds for making a variety of explosive charges such as shape charges, linear shape charges, and other types of hollow charges for demolition operations, or for making anti-tank charges. It can also be used in making mortar shells, artillery shells, missile warheads, and rocket warheads. The uniqueness of 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium makes it very useful in making high performance rocket fuels when admixed with ammonium perchlorate and ignited using a nitrocellulose ignition system. Even though 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium is non-flammable at ordinary temperatures, it burns very hot when heated above 200 Celsius in oxygen, hence when admixed with ammonium nitrate, or any strong oxidizing agent (except potassium permanganate), it deflagrates violently, and hence can be used in making high performance gun propellants; the only draw back to this is they are not smokeless in nature. In any sense, 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium is a very special and unique explosive with properties that definitely warrant further investigation; also, 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium has an acidic hydrogen attached to the ring and hence, it reacts with strong bases and alkali metals forming primary explosives that easily detonate, and these primary explosives are very useful in making priming charges for blasting caps and detonators; thus 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium can be manufactured strictly for this purpose if desired.

Safety Info: Wear gloves and use proper ventilation when handling toluene and avoid inhalation of the fumes, as toluene is a known carcinogen. Use proper ventilation and avoid inhalation of ammonia gas, which is very irritating to the nose, throat, and mucous membranes. Extinguish all flames and sources of ignition when handling acetone, hexane and diethyl ether, all three of which are highly volatile and flammable. Wear gloves when handling acetic anhydride and glacial acetic acid, and avoid skin contact. Use caution when handling 90% nitric acid, and wear gloves and use proper ventilation when handling as 90% nitric acid emits toxic fumes of nitrogen oxides.

Materials:

1. 750 milliliters of toluene	8. 450 milliliters of hexane
2. 169 grams of aluminum nitride	9. 500 milliliters of acetic anhydride
3. 5 grams of palladium foil	10. 350 milliliters of glacial acetic acid
4. Xenon gas from tank	11. 134 grams of ammonium nitrate
5. 106 grams of ammonia gas	12. 190 milliliters of 90% nitric acid
6. Oxygen gas from tank	13. 1500 milliliters of ice/salt water
7. 500 milliliters of acetone	14. 450 milliliters of dry diethyl ether

Reaction Equation:

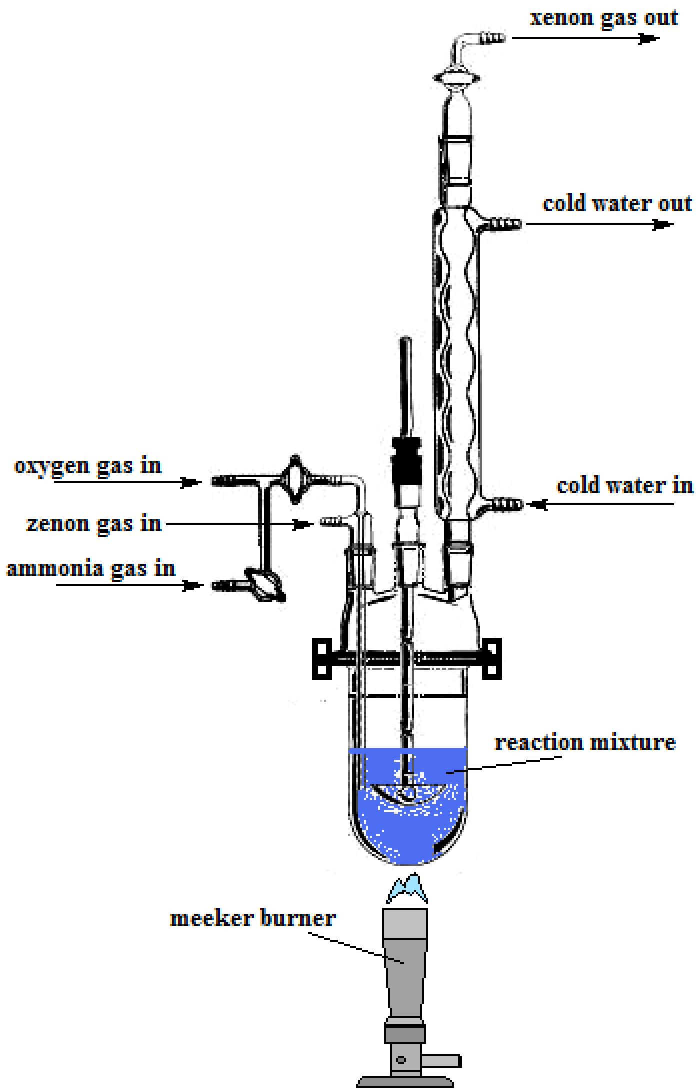


1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium

Preparation:

Step 1: Preparation of 1,3-diamino-1,3,4,5-dialuminatriazole-1,3-diium

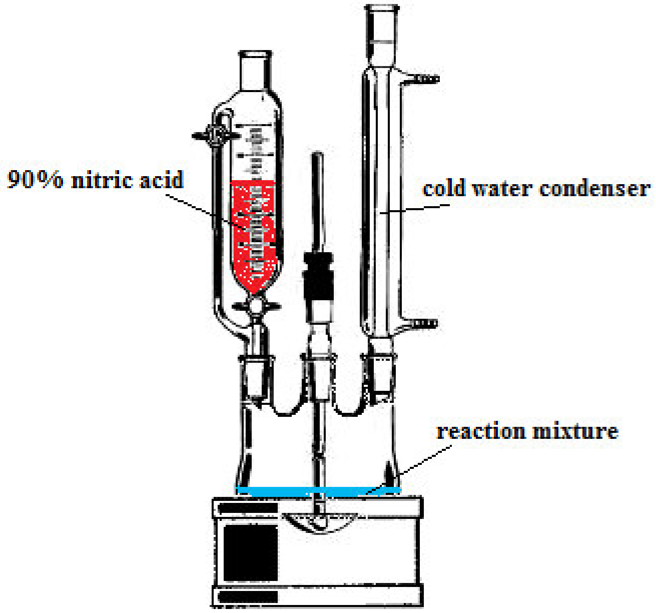
Into an apparatus as illustrated below, place 750 milliliters of toluene, followed by 169 grams of aluminum nitride, and then followed by 5 grams of palladium foil, and then begin a xenon gas purge of the apparatus. Thereafter, turn on the meeker burner and heat the contents in the reaction vessel to 350 Celsius. When the temperature of contents in the reaction vessel reaches 350 Celsius, begin to bubble into the reaction mixture dry oxygen gas at a flow rate of 100 mg per/minute, and at the same time begin to bubble into the reaction mixture dry ammonia gas at a flow rate of 150 mg per/minute while rapidly stirring the reaction mixture under reflux conditions. Continue to bubble into the reaction mixture oxygen gas and ammonia gas until 106 grams of ammonia gas has been added. During the addition of the ammonia and oxygen gases, continue to reflux the reaction mixture at 350 Celsius under rapid agitation. Once 106 grams of ammonia gas has been added, stop the ammonia gas addition, but continue to bubble into the reaction mixture dry oxygen gas for 45 minutes while continuing to reflux the reaction mixture at 350 Celsius under rapid agitation. After 45 minutes turn off the oxygen addition, and then continue to purge the apparatus with xenon gas and continue to reflux the reaction mixture at 350 Celsius under rapid stirring for 4 hours. After 4 hours, stop the xenon gas addition, and turn off the meeker burner and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to rapidly stir the reaction mixture. Thereafter, disassemble the apparatus, and then pour the reaction mixture into a large clean beaker, and then add in 500 milliliters of acetone and then stir this new mixture for 40 minutes Note: use a pair of tweezers to pick out the palladium foil pieces. Finally, filter-off the insoluble desired product of 1,3-diamino-1,3,4,5-dialuminatriazole-1,3-diium using suction filtration, and then wash with three 150-milliliter portions of dry hexane, and then suction dry as best as possible. Then save the dried washed desired product for step 2.



Setup for preparing 1,3-diamino-1,3,4,5-dialuminatriazole-1,3-diium.

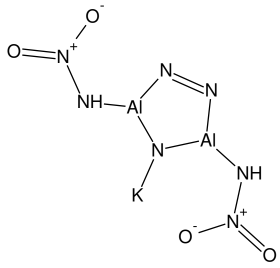
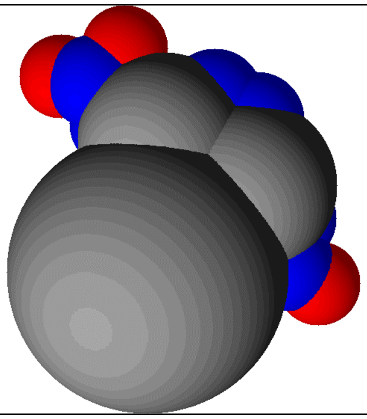
Step 2: preparation of 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium

Into an apparatus similar to one illustrated below, place 500 milliliters of acetic anhydride, followed by 350 milliliters of glacial acetic acid, followed by 134 grams of ammonium nitrate, and then followed by the 1,3-diamino-1,3,4,5-dialuminatriazole-1,3-diium prepared in step 1. Thereafter stir the mixture to form a uniform mixture. Now, pour into the addition funnel 190 milliliters of 90% nitric acid, and then turn on the heating mantle and heat the reaction mixture to 95 Celsius under rapid stirring. When the reaction mixture reaches 95 Celsius, begin a moderate drip of the 90% nitric acid into the reaction mixture while continuing to heat the reaction mixture to 95 Celsius under rapid stirring. After the addition of the 90% nitric acid, continue to stir the reaction mixture at 95 Celsius under rapid stirring for 6 hours. After 6 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during the cool down period continue to rapidly stir the reaction mixture. Thereafter, disassemble the apparatus, and pour the reaction mixture into a large beaker filled with 1500 milliliters of ice/salt water, and then stir this new mixture for 1 hour. After 1 hour, quickly filter-off the insoluble desired product of 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium using suction filtration, and then wash with three 150-milliliter portions of dry diethyl ether, and then suction dry as best as possible. Finally, place this dried filtered-off product into an oven and bake at 50 Celsius for 4 hours. After 4 hours, turn off the oven, and allow the desired product to cool to room temperature. Thereafter, place this desired product of 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium into a dessicator over concentrated sulfuric acid until use.



Setup for nitrating 1,3-diamino-1,3,4,5-dialuminatriazole-1,3-diium.

Theoretical Preparation 32: 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium; Nitramine aluminatriazole potassium salt; BFG-21

 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_2Al_2KN_7O_4$
Formula Weight = 257.1217572
Composition = H(0.78%) Al(20.99%) K(15.21%) N(38.13%) O(24.89%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 256.94361 Da
Nominal Mass = 257 Da
Average Mass = 257.1218 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6845	Non-flammable; explodes when heated	Sensitive to shock, heat, sparks, flame, percussion, pressure, and friction. Easily detonates when heated or touched by a spark or flame. A primary explosive.	Hydrolyzed by water, but generally non-reactive. Reacts with the halogens, but explodes in contact with fluorine. Oxidized by strong oxidizing agents, and will explode if grinded with potassium permanganate.	White orthorhombic crystals, or white powder or granules. Melting point: explodes; Boiling point: explodes. Insoluble in water, and hydrolyzed by it. Insoluble in most organic solvents, but soluble in liquid ammonia and sulfur dioxide.

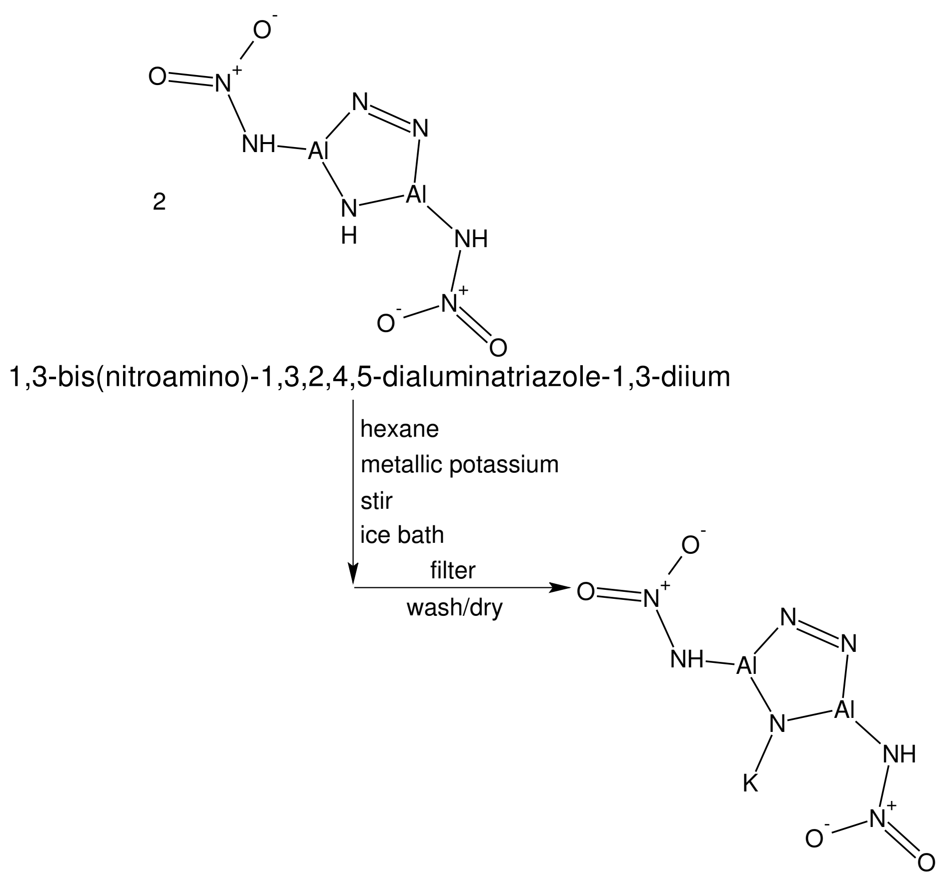
Estimated Compound summary: 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium is the potassium salt of 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium, as expected is a primary explosive. The compound can be used in primers for bullets as it explodes when struck with a firing pin or anvil. A small sample will easily detonate if grinded in mortar and pestle, or struck by a hammer. 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium is an excellent primary explosive that can be used to replace lead styphnate or lead azide in blasting caps or detonators; however, because it hydrolyzes in water this can pose a problem, and its use in bullet primers, blasting caps, or detonators, these devices would have to be sealed water-tight; if sealed in these devices water tight it can be stored and will last for many years without any type of deteriation as it would have a very long shelf life. Although this compound is more sensitive then lead azide, it is a worthy replacement for lead azide for use in many munitions; it's stability however, is great enough that it can be used in detonators for use in high velocity projectiles such as tank fired projectiles but in this case it should be diluted with dextrose. 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium can also be used in fuses for mortar rounds, and artillery shells, but it can only be used in soft target mortar and artillery fuses. It can also be use to prime detonating cord, and a variety of explosives for use in demolitions. 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium makes for a very useful all around primary explosive, which has a higher detonating velocity then other primary explosives, and its two nitramine groups gives the explosive its explosive power. It should be noted that this compound should not be used in large quantities due to its sensitivity and explosive power, and it should only be used in small quantities. 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium if desired, can be diluted with dextrose to warrant it more stable, but it still should only be used in small quantities. 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium can be used to make fireworks of the fun snaps type as it explodes when mixed with sand particles, wrapped in paper, and then thrown onto a hard surface producing a tremendous bang.

Safety Info: Extinguish all flames and sources of ignition when handling hexane, which is highly volatile and flammable. Use extreme care when handling metallic potassium; avoid contact with water, as it explodes. Wear gloves when handling metallic potassium, and avoid skin contact,

Materials:

1. 1350 milliliters of dry hexane	3. 28 grams of metallic potassium
2. 157 grams of 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium (prepared in theoretical preparation 31, step 2)	

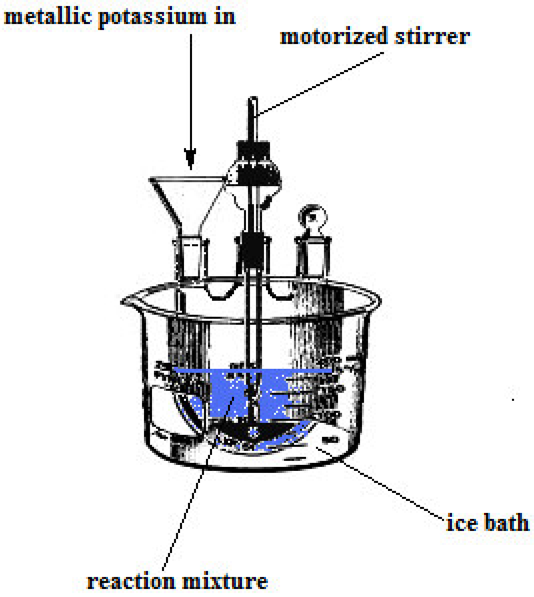
Reaction Equation:



1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium

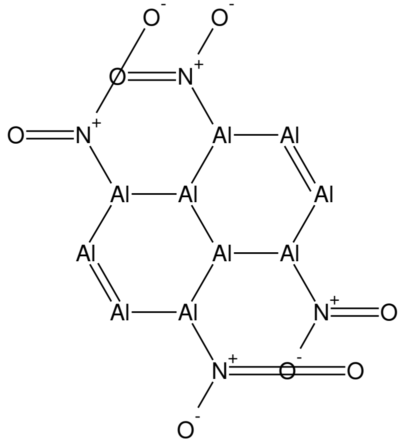
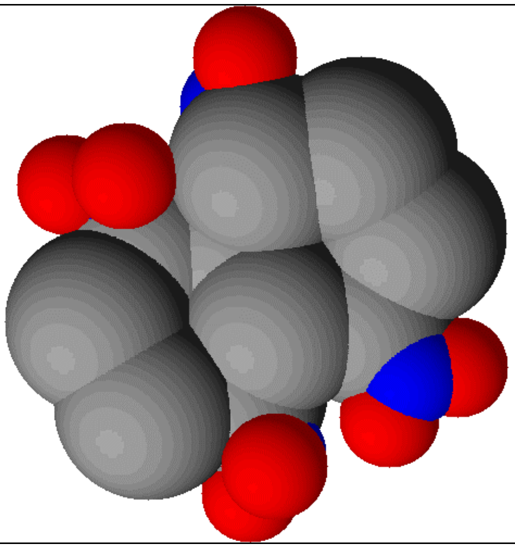
2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium

Preparation: Into an apparatus as similar to the one illustrated below pour in 600 milliliters of dry hexane, followed by 157 grams of 1,3-bis(nitroamino)-1,3,2,4,5-dialuminatriazole-1,3-diium (prepared in theoretical preparation 31, step 2). Then stir the ingredients to form a uniform mixture. Thereafter, place the flask containing these ingredients into an ice bath and chill to 5 to 10 Celsius, when the contents of the flask reach a temperature of 5 to 10 Celsius, begin to stir the reaction mixture vigorously, and then add in, in small portion at a time (100 mg portions per) of metallic potassium while keeping the temperature of the reaction mixture around 5 to 10 Celsius. Keep adding in metallic potassium until 28 grams of metallic potassium has been added. After the addition of the metallic potassium, continue to stir the reaction mixture at a temperature of 5 to 10 Celsius for 6 hours. Note: during this time, the ice will have to be replaced with fresh ice. After 6 hours, remove the ice bath, and then allow the reaction mixture to warm to room temperature. Thereafter, filter-off the insoluble desired product of 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium using suction filtration, and then wash with three 250-milliliter portions of dry hexane, and then suction dry as best as possible. Finally place the suction dried desired product onto a shallow pan, and allow it to air dry for 6 days. After 6 days, the product is ready for use.



Setup for making 2-potassium-1,3-bis(nitroamino)1,3,2,4,5-dialuminatriazole-1,3-diium.

Theoretical Preparation 33: 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum; Nitro cyclo aluminum; Al-09

 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $Al_{10}N_4O_8$

Formula Weight = 453.837386

Composition = Al(59.45%) N(12.34%) O(28.20%)

Molar Refractivity = Not available

Molar Volume = Not available

Parachor = Not available

Index of Refraction = Not available

Surface Tension = Not available

Density = Not available

Dielectric Constant = Not available

Polarizability = Not available

Monoisotopic Mass = 453.786993 Da

Nominal Mass = 454 Da
Average Mass = 453.8374 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7690; estimated by infrared spectroscopy	Non-flammable. Will burn in excess air when heated to 300 Celsius and ignited using a flame.	Non-sensitive to shock, percussion, heat, pressure, and friction. Very stable. A secondary explosive. Requires blasting cap or detonator for initiation.	Amazingly stable. Reacts with the halogens in the usual manner. Explodes in contact with fluorine. Is oxidized by strong oxidizing agents. Only very slowly hydrolyzed by water.	Bright red. Crystalline plates, or light red powder or granules. Melting point: 156 Celsius. Boiling point: decomposes when heated to 400 Celsius. Insoluble in water, and slowly hydrolyzed by it. Insoluble in all known solvents, but slightly soluble in liquid ammonia.

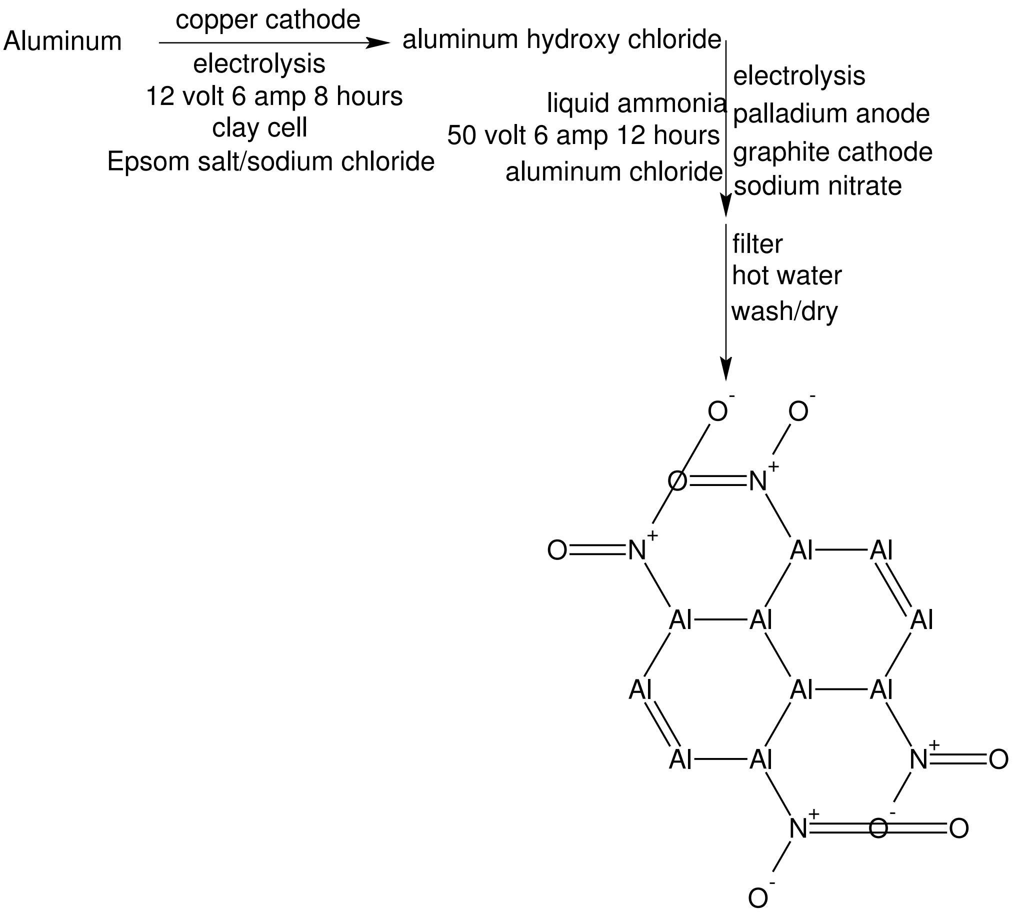
Estimated Compound summary: 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum is a remarkable cyclic aluminum explosive. The explosive has a high melting point making it useful for use in making high velocity projectiles, space based weapons, and any desired munition that may encounter heat. 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum is remarkably stable and can be stored without premature decomposition or detonation for years; however, it must be used in water-tight munitions as it has a tendency to hydrolyze in the presence of moisture or water. One interesting effect 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum has when it detonates in presence of air, the aluminum atoms within the molecule tend to bond with oxygen liberating excessive heat; hence, the compound can be used as a secondary incendiary agent that can cause secondary damage to a target. This effect makes 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum perfect for anti-tank weapons, as upon detonation the aluminum will burn in oxygen inside the tank causing excessive secondary damage and carbonizing any personnel inside the tank/vehicle. In the usual manner 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum can be melted down, and poured into molds for making a variety of shape charges both vertical and horizontal charges, poured in to mortar shells, and filling artillery shells. Due to its ability to slowly react with water, it should not be used in making land mines, torpedoes, or any other explosive munition that will be exposed to poor weather environments for prolonged periods of time.

Safety Info: Extinguish all flames before handling hexane, which is highly flammable. Use care when handling anhydrous aluminum chloride, which reacts vigorously with water.

Materials:

1. 105 grams of Epsom salt	4. 750 milliliters of hexane
2. 50 grams of sodium chloride	5. 100 grams of anhydrous aluminum chloride
3. 20 grams of aluminum foil	6. 167 grams of sodium nitrate

Reaction Equation:

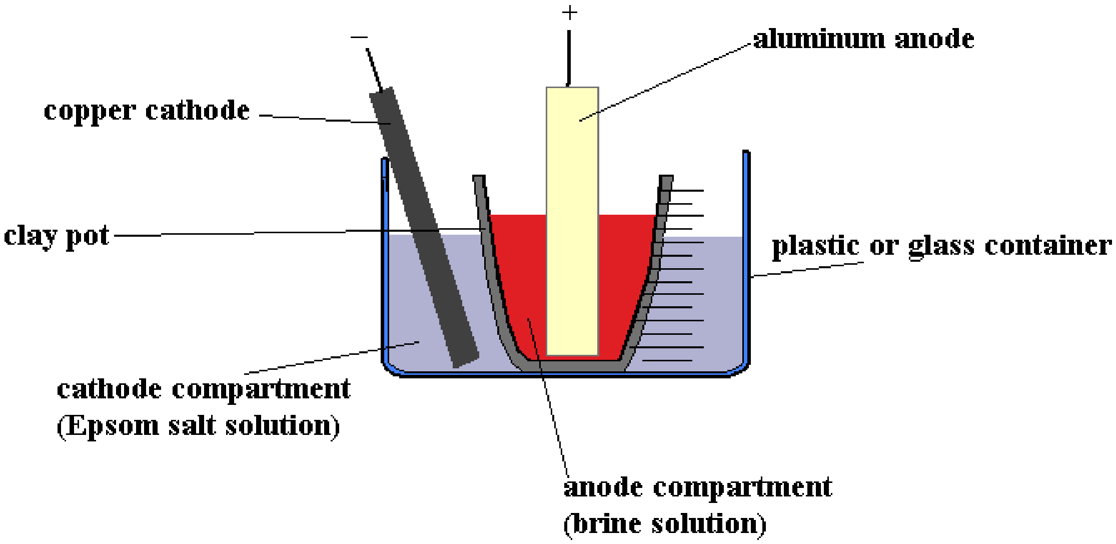


1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum

Preparation:

Step 1: Preparation of aluminum hydroxy chloride

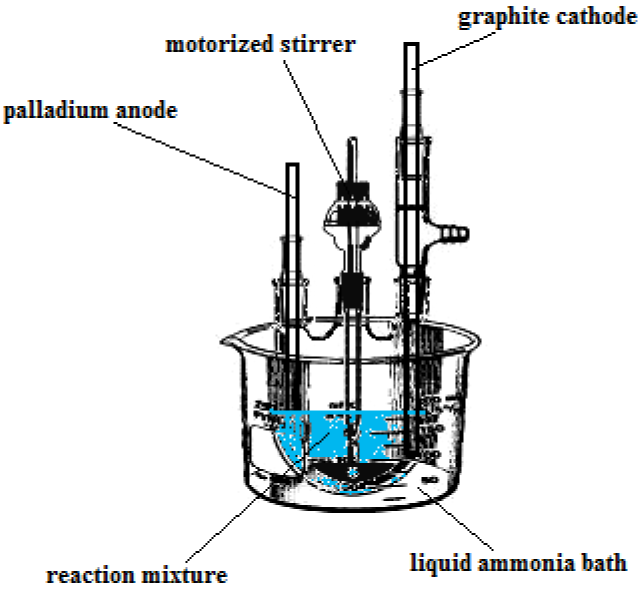
Into a clay pot cell, as illustrated below, dissolve 105 grams of Epsom salt into 750 milliliters of water and then pour this solution into the cathode compartment. Then dissolve 50 grams of sodium chloride into 500 milliliters of water, and then pour this solution into the anode compartment. Next, take 20 grams of aluminum foil, and fold into a rectangle, and this will act as the anode. Then place a copper pipe in the cathode compartment, and this will act as the cathode. Now, attach the red positive clamp to the aluminum foil anode, and attach the black negative clamp to the copper cathode. Then turn on the power supply and electrolysize the cell at 12 volt 6 amp for 8 hours. Note: during the electrolysis process, magnesium hydroxide will precipitate in the cathode compartment, and the aluminum anode will corrode. After electrolysizing the cell for 8 hours, turn off the power supply, and disconnect the negative and positive clamps. Thereafter, discard the cathode liquid, and then filter the anode liquid using suction filtration to remove insoluble impurities. Thereafter, place the filtered anode liquid into a rotary evaporator, and evaporate-off the water under high vacuum and at a temperature below 60 Celsius until dry solid remains. Note: heat will cause the desired product to decompose. Once all the water has been evaporated, and dry solid remains, stop the evaporation process, and recover the desired product of aluminum hydroxy chloride, and save for step 2.



Setup for preparing aluminum hydroxy chloride.

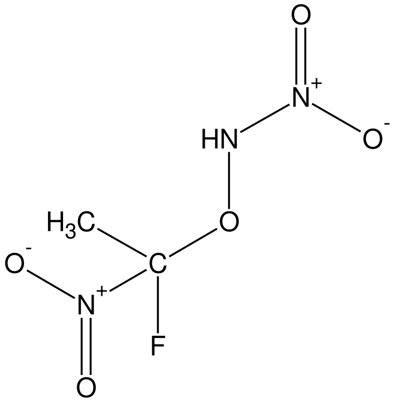
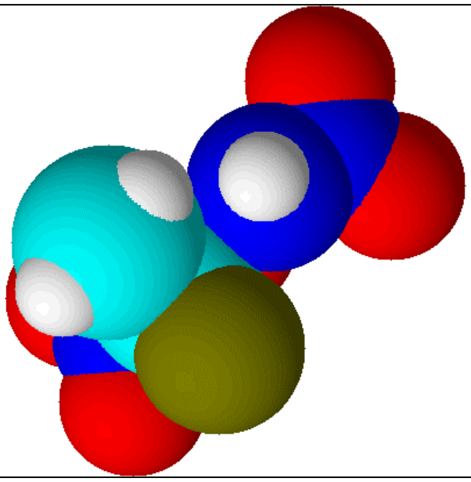
Step 2: Preparation of 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum

Into an apparatus as illustrated below, pour in 750 milliliters of hexane, followed by 100 grams of anhydrous aluminum chloride, followed by the product obtained in step 1, followed by 167 grams of sodium nitrate. Thereafter, place the reaction mixture into a liquid ammonia bath, and then place the red clamp on the palladium anode, and then place the black clamp on the graphite cathode. Thereafter, turn on the power supply at 50 volt 6 amp, and electrolysize the reaction mixture for 12 hours while vigorously stirring the reaction mixture. Note: note during the electrolysis process the desired product will form. After 12 hours time, turn off the power supply, and remove the black and red clamps. Note: during the electrolysis process, more liquid ammonia will have to be added due to evaporation of the liquid ammonia (use a fume hood with good ventilation). Thereafter, remove the apparatus from the liquid ammonia bath, and then disassemble the apparatus, and then filter-off the insoluble mass using suction filtration. Now, wash this filtered off mass with three 500-milliliter portions of hot water using caution as anhydrous aluminum chloride reacts vigorously with water. Thereafter, wash with three 500-milliliter portions of ice cold water, and then suction dry the washed material which will contain high purity desired product of 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum. Finally, place the suction dried washed desired product and place it into an oven, and bake at 105 Celsius for 4 hours. After 4 hours, turn off the oven, and then allow the desired product to cool to room temperature before removing it. Then remove the desired product and store it in a dessicator over concentrated sulfuric acid until use.



Setup for preparation of 1,4,5,8-tetranitro-1,4,4a,5,8,8a-hexacycloaluminum.

Theoretical Preparation 34: (1-fluoro-1-nitroethoxy)nitroamine; FNRN; Fluoroethoxynitramine

 (1-fluoro-1-nitroethoxy)nitroamine	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_2H_4FN_3O_5$

Formula Weight = 169.0686632

Composition = C(14.21%) H(2.38%) F(11.24%) N(24.85%) O(47.32%)

Molar Refractivity = 28.82 ± 0.3 cm^3

Molar Volume = 107.7 ± 3.0 cm^3

Parachor = 281.0 ± 4.0 cm^3

Index of Refraction = 1.447 ± 0.02

Surface Tension = 46.2 ± 3.0 dyne/cm

Density = 1.568 ± 0.06 g/cm^3

Dielectric Constant = Not available

Polarizability = 11.42 ± 0.5 $10^{-24}cm^3$

Monoisotopic Mass = 169.013498 Da

Nominal Mass = 169 Da

Average Mass = 169.0687 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6720	Flammable solid; burns with a dull green flame.	Non-sensitive to shock, percussion, heat, pressure, or friction. Very stable. A secondary explosive. Requires blasting cap or detonator for initiation.	Generally non-reactive; very stable, and pretty much chemically inert. Remarkably stable to water.	Glassy, clear solid mass. Melting point: 77 Celsius, Boiling point 198 Celsius under reduced pressure. Decomposes when heated above 240 Celsius. Insoluble in water, ether, acetone, hexane, but moderately soluble in methylene chloride.

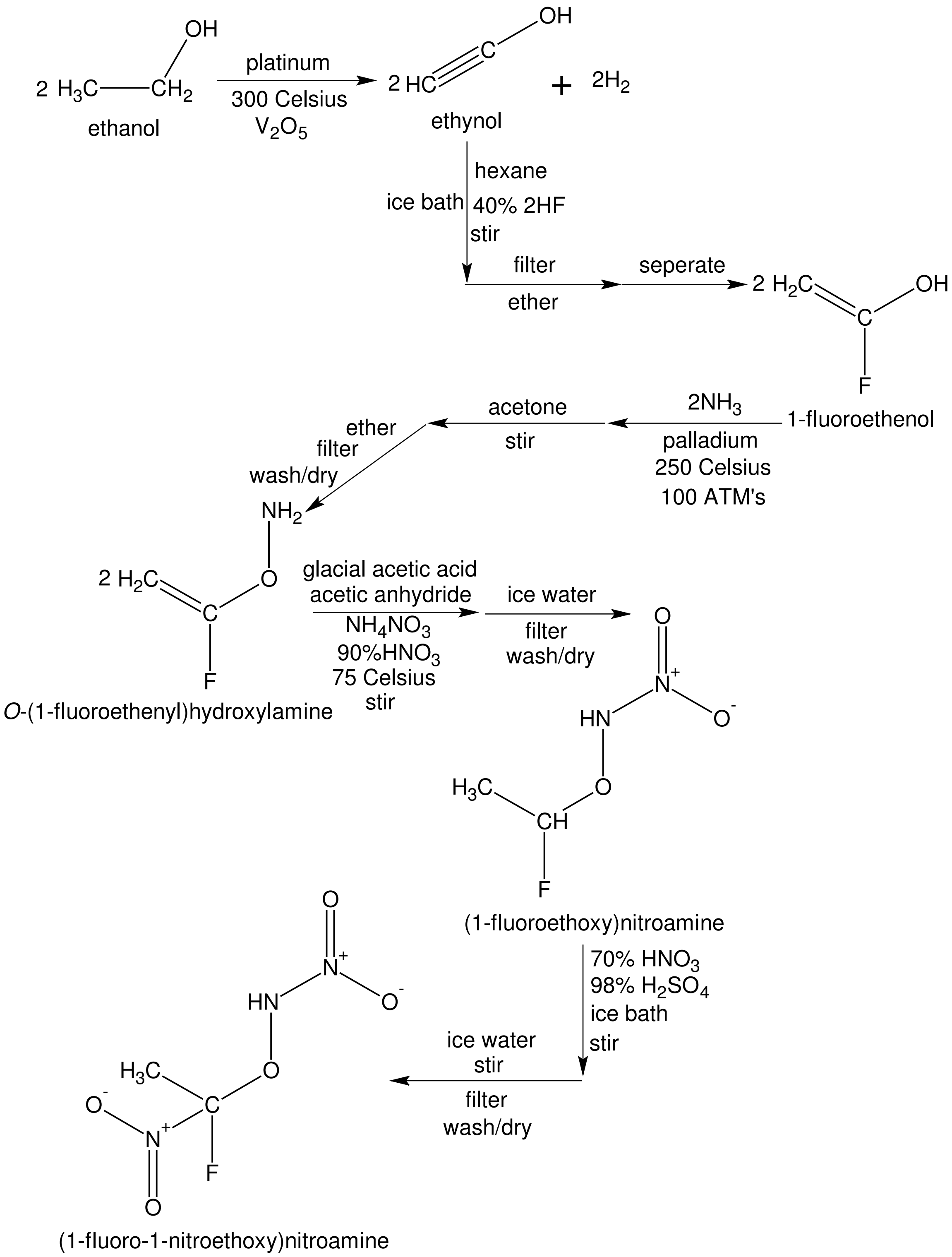
Estimated Compound summary: (1-fluoro-1-nitroethoxy)nitroamine is a remarkable new high energy plasticizer with excellent physical properties. Its low melting point and excellent thermal resistance makes it very useful for gelling nitrocellulose, and nitro starch for making blasting gels for mining operations, and for commercial and military demolition charges. It can be melted down, and mixed with other molten explosives like TNT, RDX, HMX, or PETN for making explosive compositions for a variety of commercial and military operations. The most important aspect of (1-fluoro-1-nitroethoxy)nitroamine is its ability to gel nitrocellulose for making nitroglycerine free double based high performance gun propellants for a variety of shells including rifle bullet propellants, artillery propellants, and for making propellants for propelling large shells such as battle ship guns; however, it cannot be used in making mortar propellants as its high energy upon ignition often causes the mortar tube to explode. (1-fluoro-1-nitroethoxy)nitroamine when molten, is very soluble in molten TNT and RDX, and hence can be admixed with these explosives with great ease, and then casted into molds for making shape charges, mortar bombs, artillery shells, missile warheads, and warheads for rockets. Also, the plasticizing effect of the compound is useful in making high performance rocket fuels when melted, mixed with powdered aluminum and epoxy, and then allowed to cool where upon it solidifies forming an easy to ignite high performance rocket fuel; the fact that it is first melted before and then after powdered aluminum and epoxy are added allow it to be casted into various molds for a variety of rocket designs as warranted. It should be noted, after the explosive is melted the powdered aluminum and epoxy must be thoroughly mixed within the molten explosive to avoid gaps or voids, which could lead to premature rocket failure after ignition. The great thing about this high performance rocket fuel of (1-fluoro-1-nitroethoxy)nitroamine/aluminum/epoxy is that it is a liquid before it is casted and/or allowed to solidify making it much better then common high performance rocket fuels of the ammonium perchlorate/aluminum types, as the molten (1-fluoro-1-nitroethoxy)nitroamine/aluminum/epoxy is guaranteed to avoid any cracks, gaps, holes, or air pockets when it is casted and allowed to solidify, any cracks, gaps, holes, or air pockets can cause the rocket to explode shortly after ignition.

Safety Info: Extinguish all flames and sources of ignition when handling hexane which is highly volatile and flammable. Use care when handling hydrofluoric acid, and keep it away from glass; wear gloves when handling and avoid skin contact. Use proper ventilation when handling diethyl ether and avoid inhalation of the vapors and extinguish all flames before using as diethyl ether forms explosive mixtures with air; also, never use old diethyl ether that has been on in storage for prolonged periods of time, and also perform the peroxide test before using diethyl ether. Use proper ventilation and avoid inhalation of the fumes when handling ammonia gas, which is highly irritating. Acetone is volatile and highly flammable so use caution. Wear gloves when handling glacial acetic acid and acetic anhydride and avoid skin contact. Use great care and proper ventilation when handling 90% nitric acid, which evolves toxic fumes, avoid inhalation of the fumes and wear gloves when handling. Wear gloves and avoid skin contact when handling 70% nitric acid and 98% sulfuric acid, both of which are very corrosive. Concentrated sulfuric acid is capable of charring many substances so use caution.

Materials:

1. 500 milliliters of 99% ethanol	9. 500 milliliters of glacial acetic acid
2. 750 milliliters of dry hexane	10. 750 milliliters of acetic anhydride
3. 250 grams of a 40% solution of hydrofluoric acid	11. 345 grams of ammonium nitrate
4. 3000 milliliters of dry diethyl ether	12. 190 milliliters of 90% nitric acid
5. 10 grams of palladium sponge	13. 3000 milliliters of a 10% sodium carbonate solution
6. Nitrogen gas from tank	14. 380 milliliters of 70% nitric acid
7. 85 grams of ammonia gas	15. 500 milliliters of 98% sulfuric acid
8. 800 milliliters of acetone	

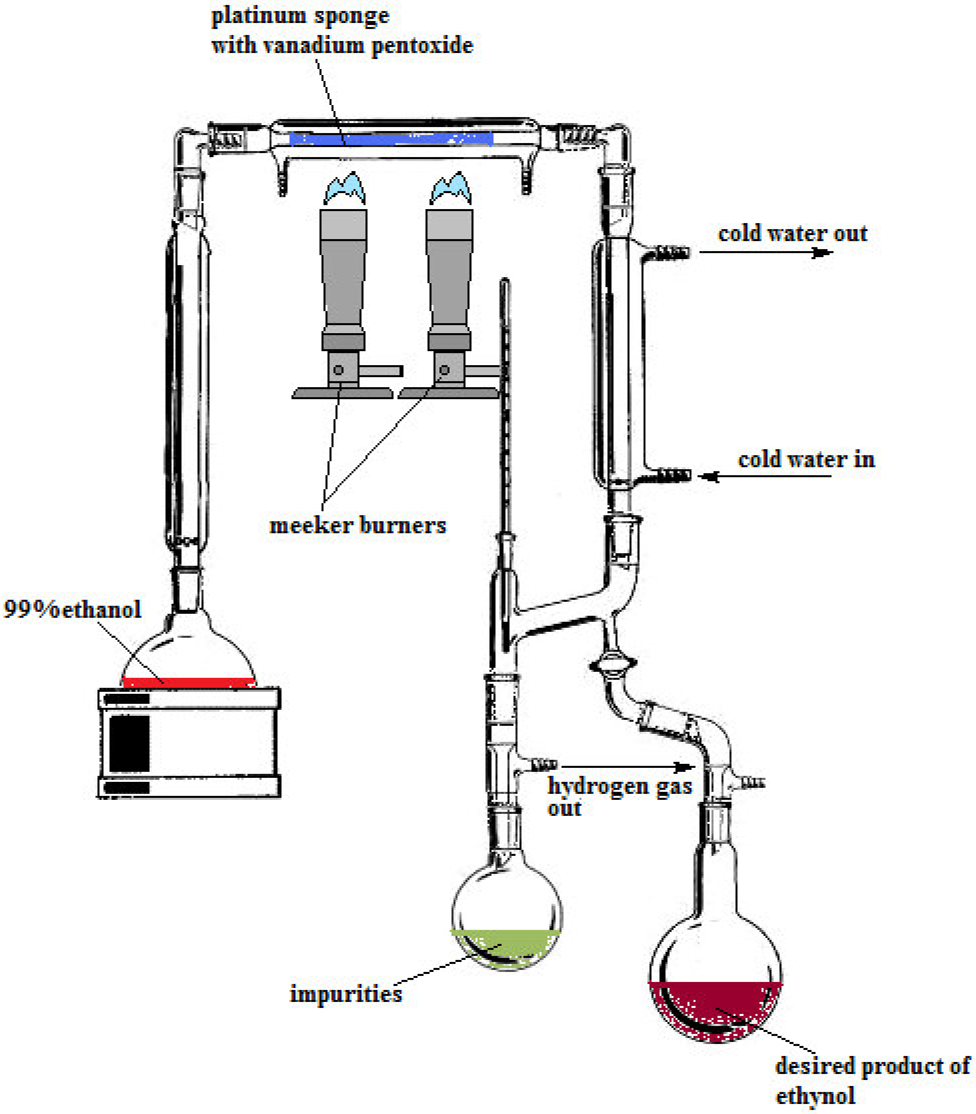
Reaction Equation:



Preparation:

Step 1: Preparation of ethynol

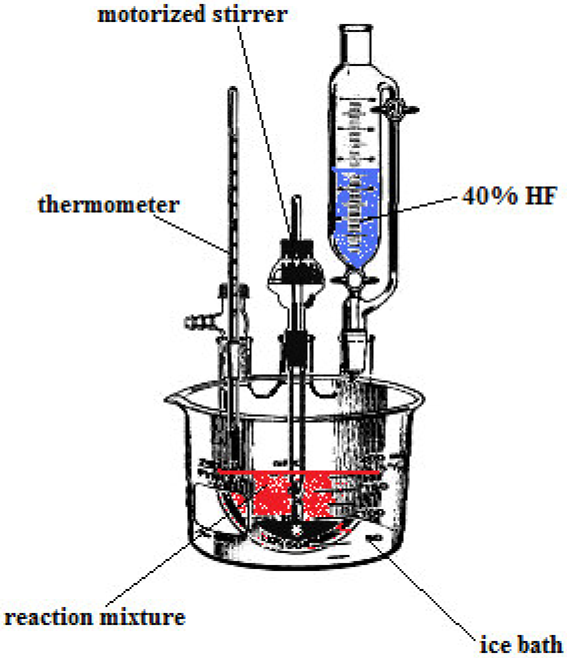
Into an apparatus as described below, place 500 milliliters of 99% ethanol into the single neck flask, and then turn on the heating mantle and heat to 120 Celsius to boil the ethanol. Shortly thereafter, ignite the meeker burners and heat the condenser filled with platinum sponge and vanadium pentoxide to 300 Celsius. Now, allow the boiling ethanol vapors to pass through the condenser containing the platinum sponge and vanadium pentoxide catalysts, during which time a chemical reaction will take place and the ethanol will be reduced and converted into the desired product of ethynol, and impurities. The impurities have a lower density, and will collect in the left receiving flask, and the ethynol will collect in the right receiver flask. Note: hydrogen gas is also a by-product so make sure the hydrogen gas does not reach the flames of the meeker burners; vent the hydrogen gas into a fume hood. Continue to boil the ethanol at 120 Celsius and continue to heat the catalysts with the meeker burners until about 3/4th of the ethanol has been boiled off and converted into the desired products. When 3/4th of the ethanol has been boiled off and converted into desired products, turn off the heating mantle and the meeker burners, and allow the apparatus to cool to room temperature. Thereafter, disassemble the apparatus and remove the right flask containing the desired liquid product of ethynol and save for step 2.



Setup for preparing ethynol.

Step 2: Preparation of 1-fluoroethanol

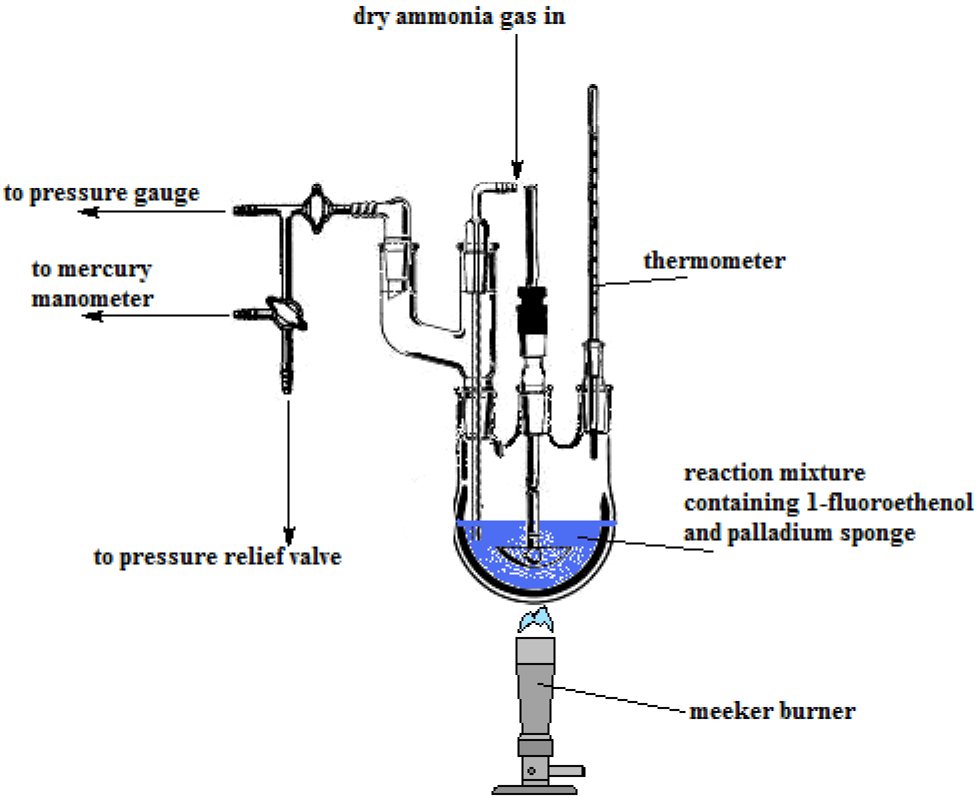
Into an apparatus as illustrated below, pour in 750 milliliters of dry hexane, and then add in 210 grams of ethynol, thereafter stir the mixture to dissolve the ethynol. Thereafter, pour 250 grams of a 40% solution of hydrofluoric acid into the addition funnel, and then place the apparatus in an ice bath as illustrated, and chill the hexane to 0 to 5 Celsius. Note: melted ice will have to be drained and fresh ice added during the reaction periodically. Now, begin to rapidly stir the reaction mixture, and then begin a slow drip of the 40% hydrofluoric acid into the hexane reaction mixture while vigorously stirring the hexane reaction mixture and keeping its temperature below 6 Celsius. After the addition of the 40% hydrofluoric acid, continue to rapidly stir the reaction mixture while maintaining its temperature below 6 Celsius at all times for 4 hours. After 4 hours, remove the ice bath and stop stirring the reaction mixture, and allow the reaction mixture to warm to room temperature. Once the reaction mixture has warmed to room temperature, disassemble the apparatus, and then filter the reaction mixture using suction filtration to remove insoluble impurities. Now, pour the filtered reaction mixture into a large beaker, and then add in 750 milliliters of dry diethyl ether, and then stir this new mixture for 1 hour. Note: the addition of the ether will cause the desired product of 1-fluoroethanol to separate out as an insoluble layer. After stirring this new mixture for 1 hour, pour the entire two-phase mixture (or break down into portions depending on how big your separatory funnel is) into a large separatory funnel, and then drain-off the lower layer carefully and collect into a clean beaker. Note: the desired product of 1-fluoroethanol will be the lower layer. In any sense, once the bottom desired product layer has been drained-off and collected, save it for step 3.



Setup for the fluorination of ethynol.

Step 3: Preparation of *O*-(1-fluoroethenyl)hydroxylamine

Into an apparatus as illustrated below, place the 1-fluoroethanol obtained in step 2, followed by 10 grams of palladium sponge. Thereafter, turn on the meeker burner, and heat this 1-fluoroethanol to around 250 Celsius with constant stirring, and bring the pressure of the reaction apparatus to 100 ATM’s by passing in dry nitrogen gas (not illustrated) until the pressure inside the apparatus reaches 100 ATM’s. Once it has, remove the nitrogen purge, and then begin to pass in dry ammonia gas into the 1-fluoroethanol reaction mixture while rapidly stirring the reaction mixture and maintaining its temperature around 250 Celsius. Continue to pass into the reaction mixture dry ammonia gas with rapid stirring until 85 grams of ammonia gas has been added. Once all the ammonia gas has been added, continue to heat and stir the reaction mixture at 250 Celsius under a pressure of 100 ATM’s for 1 hour. Note: during this 1-hour period, remove the ammonia gas flow, and replace it with dry nitrogen. After 1 hour, turn off the meeker burner, but then continue to stir the reaction mixture during the cool down period while purging the apparatus with nitrogen gas to keep the pressure inside the apparatus at 100 ATM’s during this cool down period. Once the reaction mixture has cooled to room temperature, remove the nitrogen gas purge and bring the pressure of the apparatus back to standard atmospheric pressure. Thereafter, disassemble the apparatus, and then take the 3-neck flask containing the reaction mixture (will contain a undesirable messy mass) and then add in 800 milliliters of acetone, and then using a motorized stirrer, blend this mixture rapidly for 4 hours to dissolve the desired product of *O*-(1-fluoroethenyl)hydroxylamine into the acetone. After 4 hours of stirring, stop the stirring process, remove the motorized stirrer, and then filter the mixture using suction filtration, to remove insoluble impurities, including the palladium sponge which can be removed by picking it out using tweezers. Now, to the filtered mixture pour it into a large beaker, and then pour in 750 milliliters of diethyl ether, and then stir this new mixture for 2 hours at room temperature. Note: when the ether is added, the desired product of *O*-(1-fluoroethenyl)hydroxylamine will precipitate. Finally, stop stirring, and then filter-off the precipitated desired product of *O*-(1-fluoroethenyl)hydroxylamine, wash with three 500-milliliter portions of fresh diethyl ether, and then suction dry as best as possible. Then place this suction dried mass onto a shallow pan, and allow it air-dry for 48 hours. After 48 hours, the desired product of *O*-(1-fluoroethenyl)hydroxylamine is ready for step 4.



Setup for ammoniating 1-fluoroethanol.

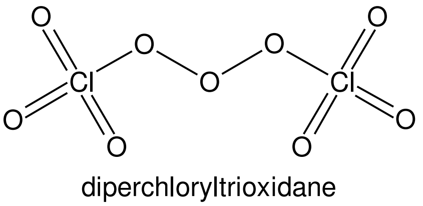
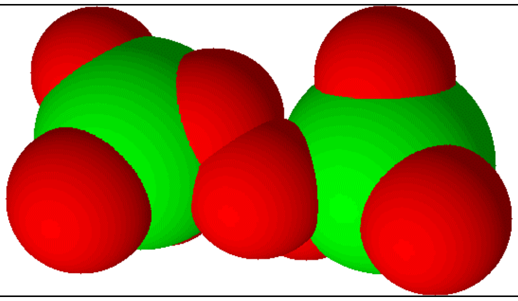
Step 4: Preparation of (1-fluoroethoxy)nitroamine

Into an apparatus including a 3-neck flask with heating mantle, addition funnel, motorized stirrer, and thermometer, place 500 milliliters of glacial acetic acid, followed by 750 milliliters of acetic anhydride, followed by 345 grams of ammonium nitrate, followed by the *O*-(1-fluoroethenyl)hydroxylamine obtained in step 3, and begin to stir this reaction mixture. Immediately thereafter, pour 190 milliliters of 90% nitric acid into the addition funnel, and then turn on the heating mantle and heat the reaction mixture to 75 Celsius with rapid stirring. When the temperature of the reaction mixtures reaches 75 Celsius, begin a slow drip of the 90% nitric acid into the reaction mixture while heating the reaction mixture at 75 Celsius under rapid agitation. Once all the 90% nitric acid has been added, continue to heat the reaction mixture to 75 Celsius under rapid stirring for 4 hours. After 4 hours, continue to rapidly stir the reaction mixture, but turn off the heating mantle and allow the reaction mixture to cool to room temperature. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a large beaker filled with 2000 milliliters of ice water, and then stir this new mixture for 6 hours. After 6 hours, filter-off the insoluble desired product of (1-fluoroethoxy)nitroamine using suction filtration, and then wash this filtered-off product with three-500 milliliter portions of hot water, followed by washing with three 500-milliliter portions of a 10% sodium carbonate solution, and then finally wash with three 500-milliliter portions of hot water, and then suction dry this washed product using suction filtration as best as possible, and then save this suction dried product for step 5.

Step 5: Preparation of (1-fluoro-1-nitroethoxy)nitroamine

Into a suitable sized beaker, equipped with a motorized stirrer, thermometer and ice bath, place 380 milliliters of 70% nitric acid, and then slowly and carefully add in 500 milliliter of 98% sulfuric acid. Once the sulfuric acid has been added, wait until this nitrating acid mixture has chilled to 5 Celsius, and then begin stirring this nitrating acid and then add in, in small portions at a time, the product of (1-fluoroethoxy)nitroamine obtained in step 4 over a period of 4 hours, while vigorously stirring the reaction mixture and maintaining it temperature below 10 Celsius. Once all the (1-fluoroethoxy)nitroamine has been added, continue to stir the reaction mixture at a temperature below 10 Celsius for 6 hours. Note: the ice bath will have to be drained and refilled with ice periodically during the addition of the (1-fluoroethoxy)nitroamine. After 6 hours, remove the ice bath, and allow the reaction mixture to warm to room temperature. Thereafter, gently and slowly pour this entire reaction mixture into a super large beaker filled with 2000 milliliters of ice water, and then stir this new mixture for 2 hours. After 2 hours, filter-off the insoluble desired product of (1-fluoro-1-nitroethoxy)nitroamine using suction filtration, wash with three 500-milliliter portions of a 10% sodium carbonate solution, and then wash with three 750-milliliter portions of hot water, and then suction dry as best as possible. Finally, place this filtered suction dried product onto a shallow pan, and allow it to air-dry for 48 hours. After 48, the desired product of (1-fluoro-1-nitroethoxy)nitroamine is ready for use.

Theoretical Preparation 35: diperchloryltrioxidane; Perchloryl-T; DPT-Dane; CL-57

 O O O O O O O O O Cl Cl diperchloryltrioxidane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = Cl_2O_9
Formula Weight = 214.9006
Composition = Cl(32.99%) O(67.01%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 213.891937 Da
Nominal Mass = 214 Da
Average Mass = 214.9006 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7290; estimated using infrared spectroscopy	Non-flammable. Will ignite violently if mixed with powdered metals or reducing agents and touched by a spark or flame.	Moderately stable towards percussion, heat, pressure, shock, or pressure. A small sample will detonate when struck by a hammer. A secondary explosive. Requires blasting cap or detonator for proper initiation.	Generally non-reactive, but is slowly hydrolyzed by water forming hydrogen peroxide, chlorine monoxide, and hypochlorous acid. Reacts with hot concentrated hydrochloric acid liberating chlorine gas,	White monoclinic crystals, powder, or granules. Melting point: 88 Celsius (when slowly heated—explodes if rapidly heated); boiling point: explodes when heated to 150 Celsius. Slightly soluble in water, but slowly hydrolyzed by it, 1 grams of explosive dissolves in 9 milliliters of water

				with a half life of 12 days. Insoluble in ether, acetone, alcohol, methylene chloride, and toluene. Moderately soluble in liquid ammonia.

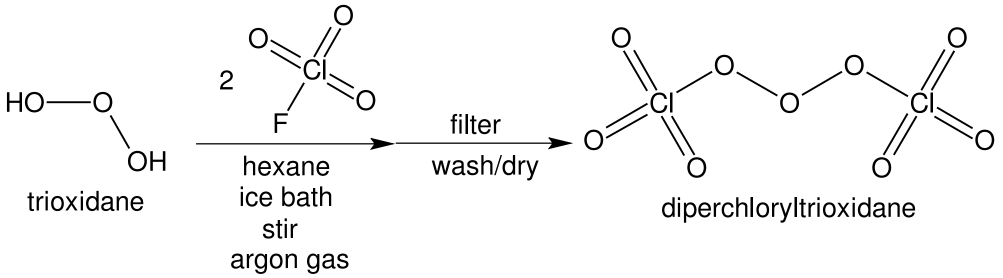
Estimated Compound summary: diperchloryltrioxidane is the chlorate derivative of trioxidane, and is a remarkable and fascinating explosive. Its two perchloryl groups has great bond energy between the oxygen atoms and the chlorine atoms, and hence when detonated the explosive gives off a powerful pressure wave. Diperchloryltrioxidane has a limited use in making mortar bombs, artillery shells, but its powerful pressure wave upon detonation makes it perfect for penetrating missile warheads, drop bombs, hand grenades, and long range rockets. Diperchloryltrioxidane can be melted down and cast into shape charges in the usual manner, however, molten diperchloryltrioxidane is insoluble in other molten explosives so its use in making explosive compositions is void; none the less, it can be melted down, and poured into molds or it can be dead pressed into tubes for use in blasting hard rock for construction purposes, and the pressure wave produced by diperchloryltrioxidane makes it a very powerful rock blaster. Because of its two perchloryl groups Diperchloryltrioxidane can be melted down and mixed with powdered aluminum and epoxy in making high performance rocket fuels for a variety of commercial and military uses—in this manner, the liquid explosive mixture can be poured into molds for a variety of rocket designs as determined. As discussed in a previous preparation, melting down the diperchloryltrioxidane and then thoroughly mixing it with powdered aluminum and epoxy and pouring it into molds for a variety of rocket designs, it voids cracks, holes, or air pockets; thereby decreasing the chance of premature rocket failure. Diperchloryltrioxidane can also be used in making high performance gun propellants when mixed with strong reducing agents. On a lighter note, diperchloryltrioxidane can be used in making fireworks, as it burns brilliantly but violently when mixed with powdered metals and reducing agents producing a large variety of color effects, or it can be used as a bursting charge in aerial shells producing tremendous bangs.

Safety Info: Extinguish all flames and sources of ignition before handling hexane, which is highly volatile and flammable. Use extreme care when handling perchloryl fluoride, and avoid skin contact, and inhalation as perchloryl fluoride is toxic; also perchloryl fluoride has a tendency to explode in contact with organic matter so use care.

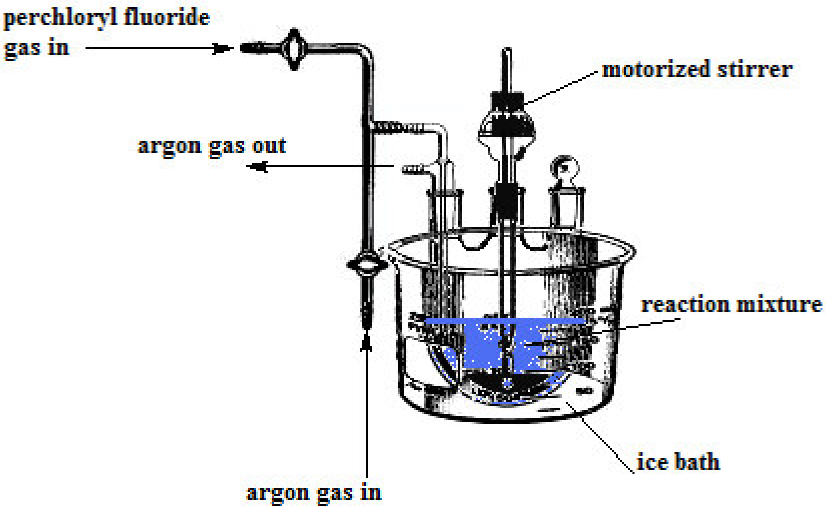
Materials:

1. 750 milliliters of hexane	4. 561 grams of perchloryl fluoride gas
2. 137 grams of trioxidane (prepared in theoretical preparation 4, step 1)	5. 1500 milliliters of a 10% sodium carbonate solution
3. Argon gas from tank	

Reaction Equation:

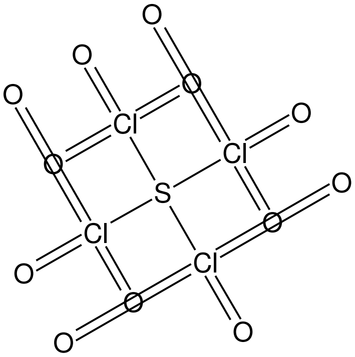
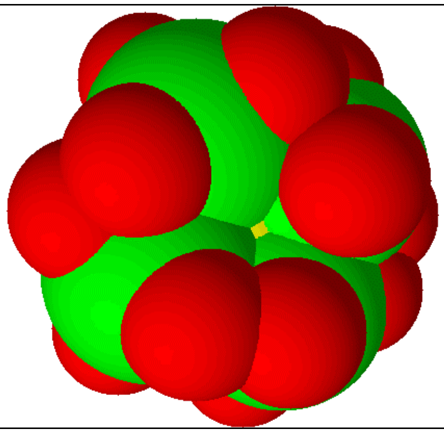


Preparation: Into an apparatus similar to the one illustrated below, place 750 milliliters of hexane, and then add in 137 grams of trioxidane (prepared in theoretical preparation 4, step 1), and then vigorously stir this mixture for 30 minutes to form a uniform mixture. Thereafter, place the apparatus into an ice bath, and chill the reaction mixture down to 5 to 0 Celsius. When the temperature of the reaction mixture reaches 5 to 0 Celsius, begin to purge the apparatus with argon gas at a flow rate of 500 mg per/minute, and then 5 minutes later, begin to bubble in dry perchloryl fluoride gas at a flow rate of 50 mg per/minute, while maintaining the reaction mixtures temperature around 5 to 0 Celsius at all times with constant stirring. Continue to bubble in argon gas and perchloryl fluoride gas at their respective flow rates until 561 grams of perchloryl fluoride gas has been added. Note: maintain vigorous stirring and cooling of the reaction mixture during the entire addition of the perchloryl fluoride gas; also the ice bath will have to be drained and re-filled with ice periodically during the entire process. After all the perchloryl fluoride gas has been added, continue to vigorously stir the reaction mixture and maintain its temperature around 5 to 0 Celsius with constant stirring for 6 hours. Note: continue to purge the apparatus with argon gas at its respective flow rate during this 6-hour period. After 6 hours, remove the argon gas purge, remove the ice bath, and allow the reaction mixture to warm to room temperature. Note: continue to vigorously stir the reaction mixture during the warming process. Once the reaction mixture has warmed to room temperature, disassemble the apparatus, and then filter-off the insoluble product of diperchloryltrioxidane using suction filtration. Thereafter, wash the filtered-off product with three 500-milliliter portions of a 10% sodium carbonate solution, followed by washing with six 500-milliliter portions of ice-cold water, and then suction dry as best as possible. Finally, place the washed suction dried product onto a shallow pan, and allow it to air-dry for 48 hours. After 48 hours collect the dried product of diperchloryltrioxidane and then store in an air-tight container until use.



Setup for preparing diperchloryltrioxidane.

Theoretical Preparation 36: λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide; Chlorane; Sulfur chlorane; λ^4perchloryl sulfide

 λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $Cl_4O_{12}S$
Formula Weight = 365.8698
Composition = Cl(38.76%) O(52.48%) S(8.76%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available

Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 363.786456 Da
Nominal Mass = 364 Da
Average Mass = 365.8698 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
4,980	Flammable in presence of oxygen only burning to form sulfur dioxide and chlorine dioxide, which often explodes shortly after ignition.	Sensitive to friction, but non-sensitive to heat, percussion, pressure, or shock. Mostly stable under most conditions. A secondary explosive. Requires blasting cap or detonator for proper initiation.	Generally non-reactive. Does not react with oxidizing agents, but it is slowly hydrolyzed by water. Hydrolyzes much more rapidly when added to boiling water, and may explode.	Yellowish amorphous solid when pure, but yellowish red when impure. Melting point: 109 Celsius; Boiling point: explodes when heated to 450 Celsius. Insoluble in water, and most organic solvents, but moderately soluble in liquid ammonia and liquid sulfur dioxide.

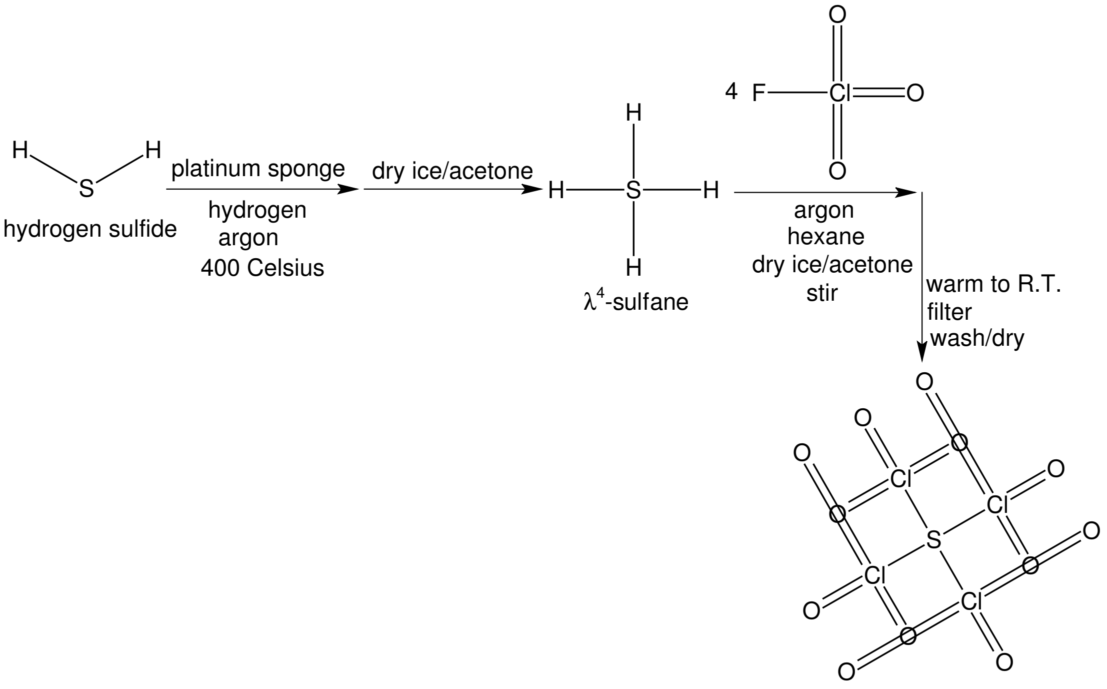
Estimated Compound summary: λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide is an interesting multi perchloryl explosive with a chiral sulfur center. The compound is sensitive to friction and hence can be used to make matches, and friction designed ignition systems. The compound is also useful in making pyrotechnic mixtures as it burns and even explodes brilliantly when mixed with powdered metals and reducing agents. λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide would primarily be used to make pyrotechnic formulations as its low detonating velocity excludes it from making any significant explosive munitions or explosive compositions; however, it can be melted down and poured into molds for use in making shape charges, mortar bombs, artillery shells, drop bombs, or missile warheads, but its cost of manufacture and low detonating velocity makes it rather undesirable in these areas—not to mention the fact that when molten, it is insoluble in most common molten explosives such as TNT, RDX, HMX, ect., ect.,; λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide when mixed with powdered aluminum and cured with epoxy makes a powerful and very desirable high performance rocket fuel for use in all types of known rockets for various purposes. Also, its thrust to weight ratio is very good, and its exhaust fumes are much lower in temperature then more common known high performance rocket fuels thereby making it cheaper to manufacture rockets for all known purposes. When λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide is mixed with powdered iron, it explodes with great brilliance when sparked or touched by a flame—this could be used as a bursting charge in aerial fireworks. When λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide is mixed with powered magnesium and cured with epoxy, it produces tremendous amounts of light 4 time greater then regular burning magnesium; hence making for a pyrotechnic composition that can be used in illumination. Amazingly enough, when λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide is mixed with powdered sulfur and/or charcoal it burns at an amazing speed 3 times faster then ordinary black powder; hence making it very useful in making gun powders for various propellant means including bullets, and shells of various types and for various purposes. All in all λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide is a fascinating explosive that shows an enormous talent for making pyrotechnic compositions, and these compositions are virtually unexplored and endless.

Safety Info: Use proper ventilation, and avoid inhalation of hydrogen sulfide, which is toxic. Extinguish all flames and sources of ignition before using hydrogen, which forms explosive mixtures with air. Also, extinguish all flames before handling hexane, which is highly volatile and flammable. Use caution when handling perchloryl fluoride gas, which is very reactive and may ignite organic material, avoid inhalation and skin contact. Use proper ventilation and avoid inhalation of diethyl ether, which is extremely volatile and forms explosive mixtures with air; also, never use old diethyl ether as peroxides may be present and will explode if heated; perform the peroxide test on any diethyl ether used before using.

Materials:

1. Argon gas from tank	4. 750 milliliters of dry hexane
2. 147 grams of hydrogen sulfide gas	5. 1770 grams of perchloryl fluoride gas
3. 10 grams of hydrogen gas	6. 1500 milliliters of diethyl ether

Reaction Equation:

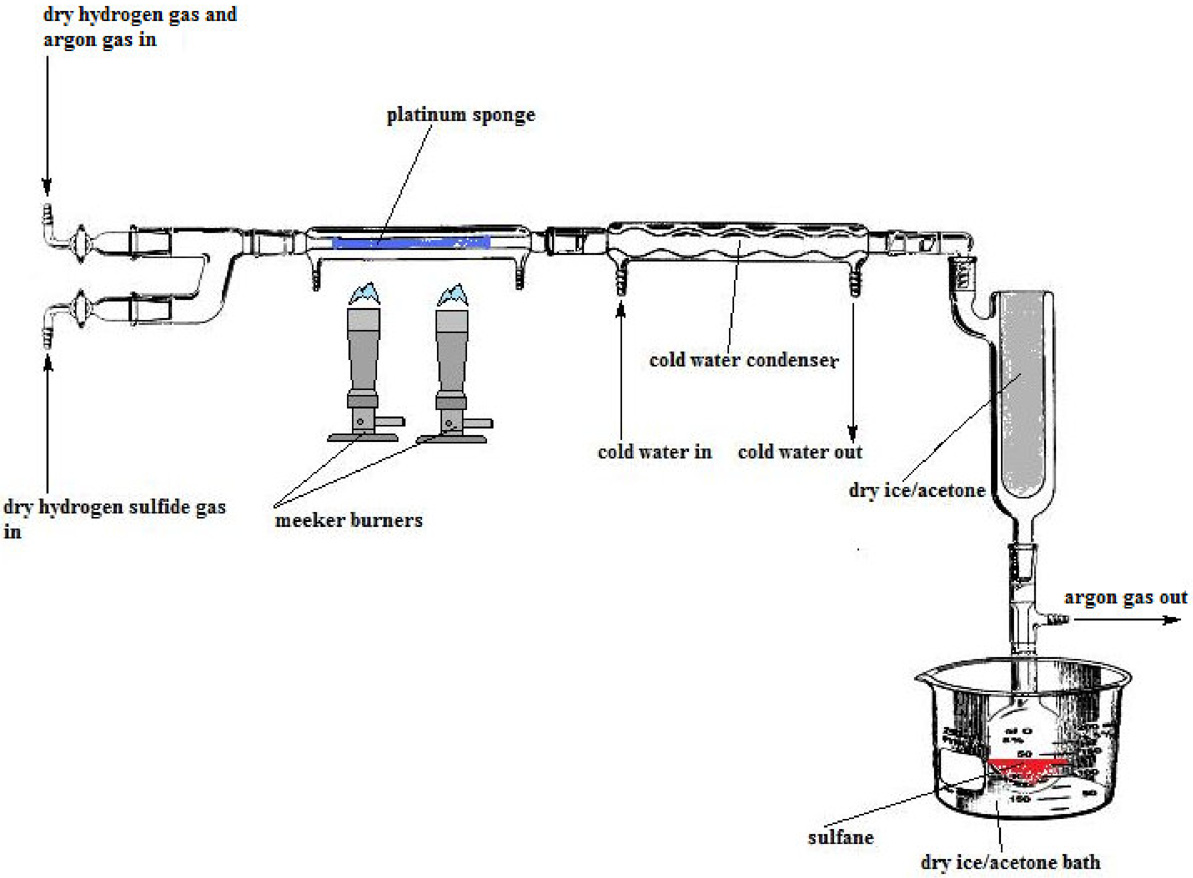


λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide

Preparation:

Step 1: Preparation of λ^4-sulfane

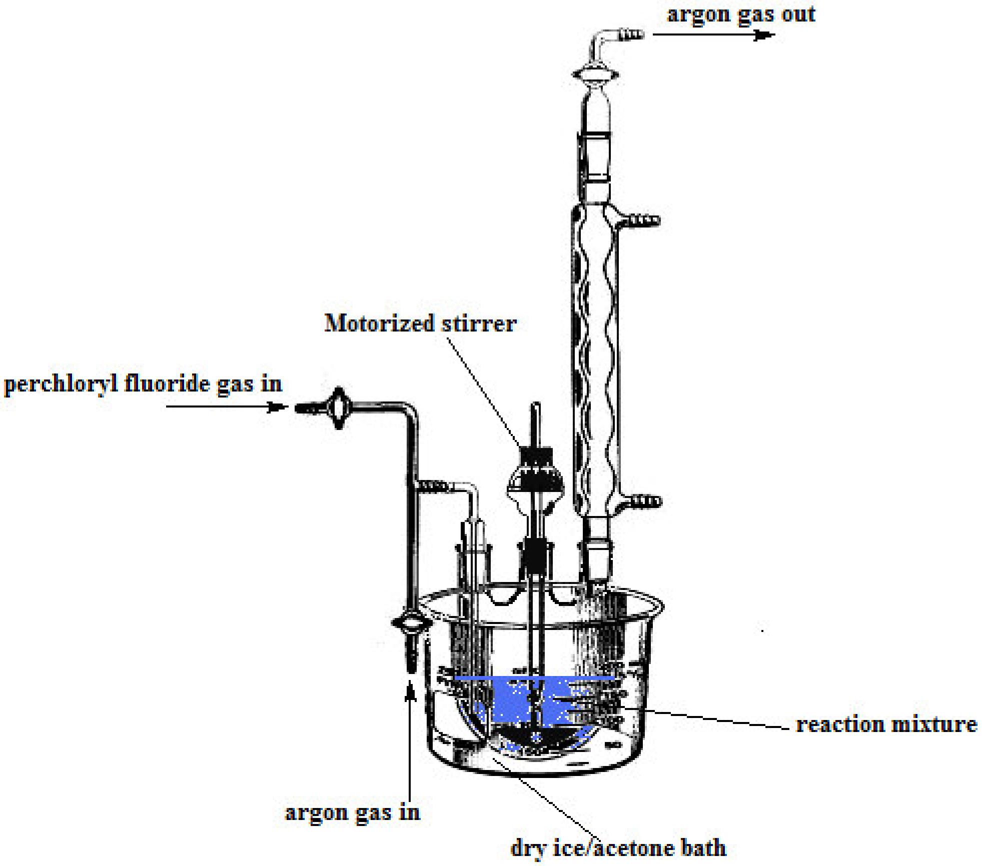
Into a special apparatus as illustrated below, purge the apparatus with dry argon to exclude air, and then turn on the meeker burners, and heat the platinum sponge to 400 Celsius. Thereafter, begin to pass into the apparatus argon at a flow rate of 500 mg per/minute, along with dry hydrogen gas at a flow rate of 50 mg per/minute, along with dry hydrogen sulfide gas at a flow rate of 750 mg per/minute. Continue to pass into the apparatus argon, hydrogen, and hydrogen sulfide at their respective flow rates until 147 grams of hydrogen sulfide gas, and 10 grams of hydrogen gas has been added. Note: λ^4-sulfane is a gas at room temperature, so we use dry ice/acetone to freeze it to a liquid. Prepare the dry ice/acetone bath by mixing 3 parts dry ice with 6 parts acetone. Once the desired amount of hydrogen gas and hydrogen sulfide gas has been added, the reaction is over, and you can turn off the meeker burners, turn off the argon gas purge, and then disassemble the apparatus, but keep the desired product λ^4-sulfane in its receiver flask chilled in a dry ice/acetone bath for step 2.



Setup for preparing λ^4-sulfane.

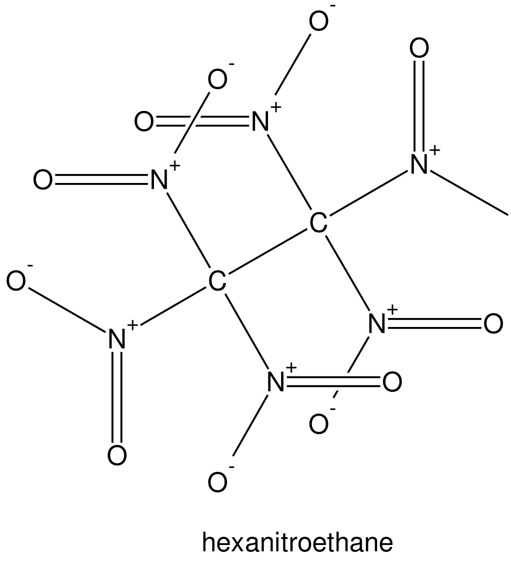
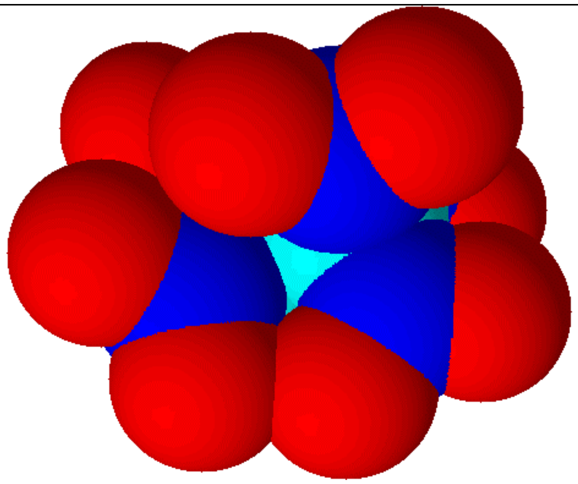
Step 2: preparation of λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide

Into an apparatus as illustrated below, add in 750 milliliters of dry hexane, followed by the λ^4-sulfane prepared in step 1, and then begin an argon purge to flush out air, and then continue to purge the apparatus with argon while bubbling in perchloryl fluoride gas at a flow rate of 750 mg per/minute while rapidly stirring the reaction mixture. Note: the dry ice acetone bath will have to be replaced with fresh dry ice during the procedure. Continue to bubble in dry perchloryl fluoride gas until a massive 1770 grams of perchloryl fluoride gas has been added. Continue to bubble in dry perchloryl fluoride gas while rapidly stirring the reaction mixture and maintaining a proper dry ice/acetone bath. After the addition of the perchloryl fluoride gas, continue to vigorously stir the reaction mixture while maintaining a fresh dry ice acetone bath for 4 hours. After 4 hours, remove the dry ice acetone bath, and allow the reaction mixture to warm to room temperature, but continue to vigorously stir the reaction mixture during this warm up period. Once the reaction mixture has warmed to room temperature, filter-off the insoluble product of λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide using suction filtration, and then wash with three 500-milliliter portions of dry diethyl ether, followed by washing with three 1500-milliliter portions of cold water, and then suction dry as best as possible. Finally, place the dried washed product of λ^4-sulfanetetrayltetrakis(chlorane) dodecaoxide into an oven and cook at 101 Celsius for 8 hours. After 8 hours, turn off the oven, and allow the desired product to cool to room temperature. Once the desired product has cooled to room temperature, store it in an airtight container until use.



Setup for perchlorination of λ^4-sulfane.

Theoretical Preparation 37: Hexanitroethane; polynitro ethane; HNE; COMP N

	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_2N_6O_{12}$

Formula Weight = 300.0544

Composition = C(8.01%) N(28.01%) O(63.99%)

Molar Refractivity = 47.38 ± 0.3 cm^3

Molar Volume = 138.3 ± 3.0 cm^3
Parachor = 453.1 ± 4.0 cm^3
Index of Refraction = 1.600 ± 0.02
Surface Tension = 115.2 ± 3.0 dyne/cm
Density = 2.169 ± 0.06 g/cm^3
Dielectric Constant = Not available
Polarizability = 18.78 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 299.957419 Da
Nominal Mass = 300 Da
Average Mass = 300.0544 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6780 to 6990 depending on purity	Non-flammable. Forms deflagrating compositions when mixed with powdered metals or reducing agents.	Extremely stable. Non-sensitive to shock, percussion, heat, pressure, or friction. A secondary explosive. Requires blasting cap or detonator for initiation.	Generally non-reactive. Can be reduced with tin and hydrochloric acid	White to colorless to light yellow crystalline mass depending on purity, melting point: 135 Celsius; boiling point: sublimes; decomposes above 400 Celsius; Insoluble in water, ether, acetone, but moderately soluble in methylene chloride, hexane, n-octane,

Estimated Compound summary: Hexanitroethane is a poly alkyl explosive used to make explosive compositions by melting it down and adding it to other molten explosives such as TNT, RDX, HMX, PETN, ect., ect.,; hexanitroethane is a typical high explosive with typical and similar characteristics as other poly nitro explosives, but it's main potential is as an oxidizer in high performance rocket propellants especially when mixed with powdered aluminum or finely divided boron and cured with epoxy. Hexanitroethane is also commonly used in pyrotechnics for making a variety of flare compositions for commercial and military uses. Other uses hexanitroethane has is for making high performance gun propellants when mixed with high performance energetic plasticizers and cured. If used by itself in making explosive munitions it can either be dead pressed into shell casings, used in the form of granules in grenades, or melted down and poured into molds for making shape charges and for filling mortar shells, and artillery shells. Hexanitroethane can also be dead pressed under 5,000 psi into missile casings for warheads, or melted down and either poured into missile casings for warheads, or mixed with other molten explosives for use in making missile warheads for a variety of uses.

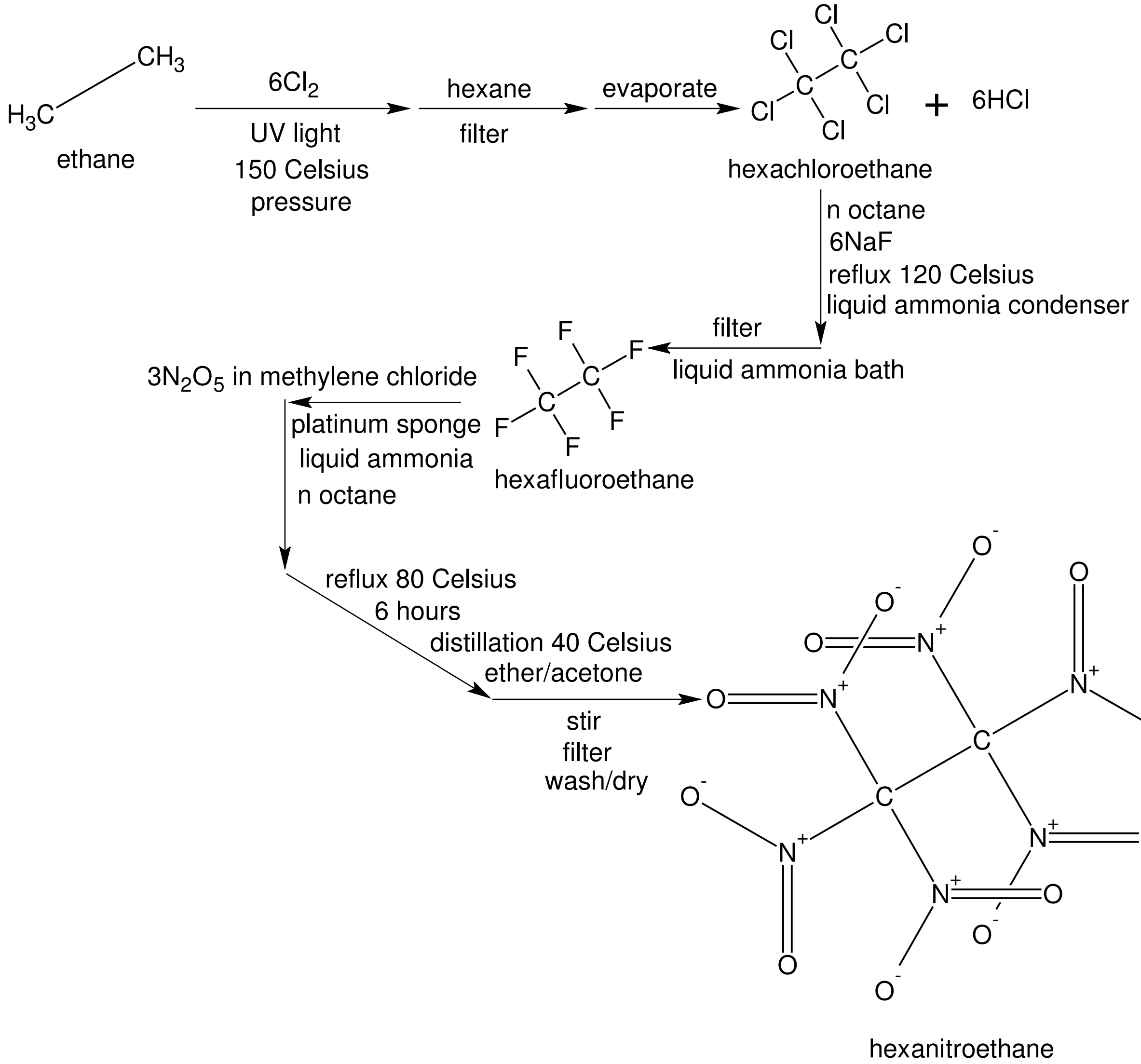
Safety Info: Extinguish all flames before using ethane, which is highly flammable. Use maximum ventilation when handling chlorine gas, which toxic so use caution. Extinguish all flames and sources of ignition before handling hexane and n-octane, both of which are volatile and highly flammable. Use caution when handling dinitrogen pentoxide, and avoid contact with organic material as fire may result. Use proper ventilation and avoid inhalation of diethyl ether, which is extremely volatile and forms explosive mixtures with air; also, never use old diethyl ether as peroxides may be present and will explode if heated; perform the peroxide test on any diethyl ether used before using. Acetone is highly flammable, so use caution.

Materials:

1. Argon gas from tank	7. 5 grams of platinum sponge
2. 109 grams of ethane gas	8. 1,194 grams of dinitrogen pentoxide

3. 1,542 grams of chlorine gas	9. 3000 milliliters of methylene chloride
4. 2500 milliliters of dry hexane	10. 1500 milliliters of diethyl ether
5. 3000 milliliters of n-octane	11. 1500 milliliters of acetone
6. 923 grams of anhydrous sodium fluoride	

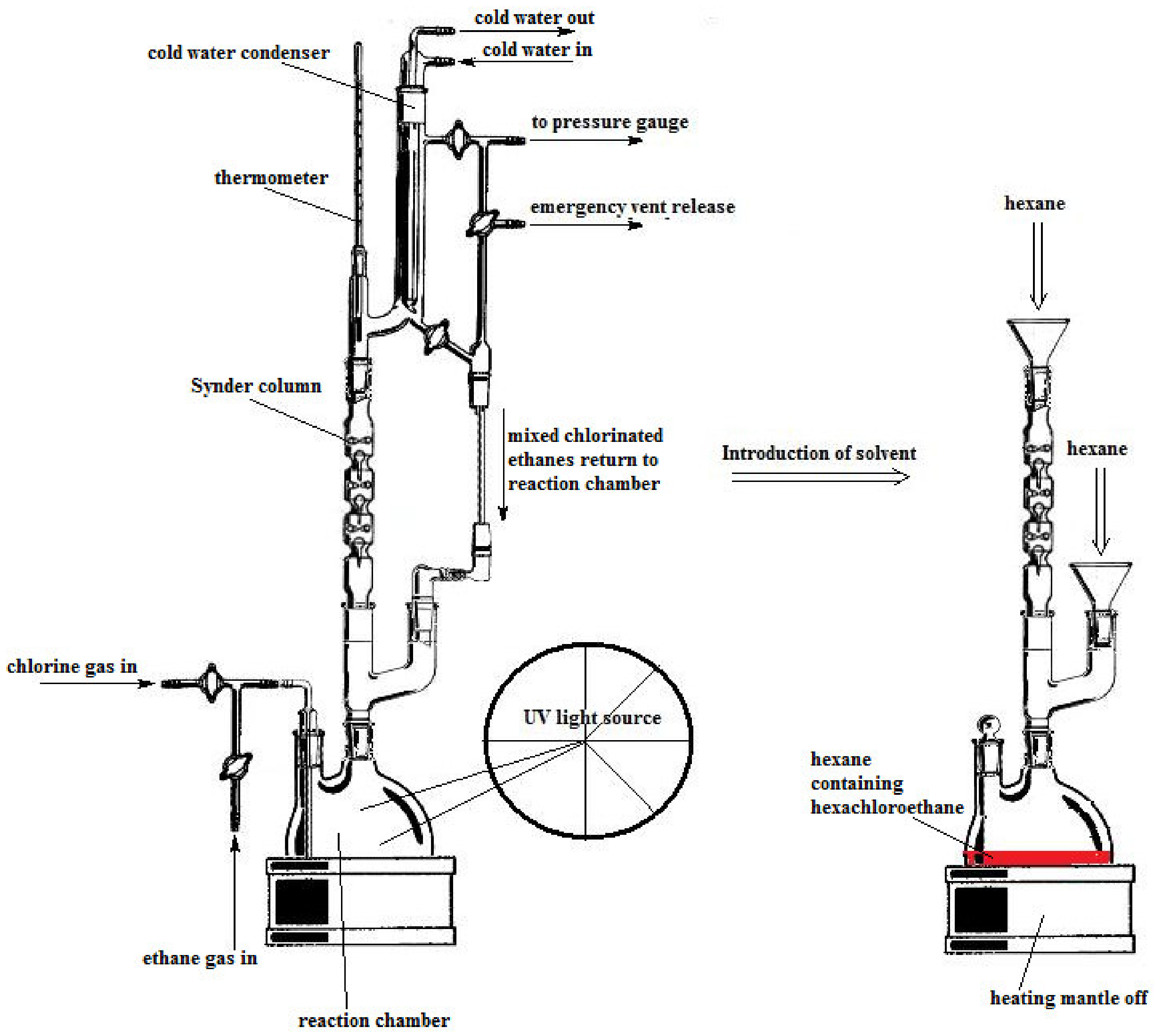
Reaction Equation:



Preparation:

Step 1: Preparation of hexachloroethane

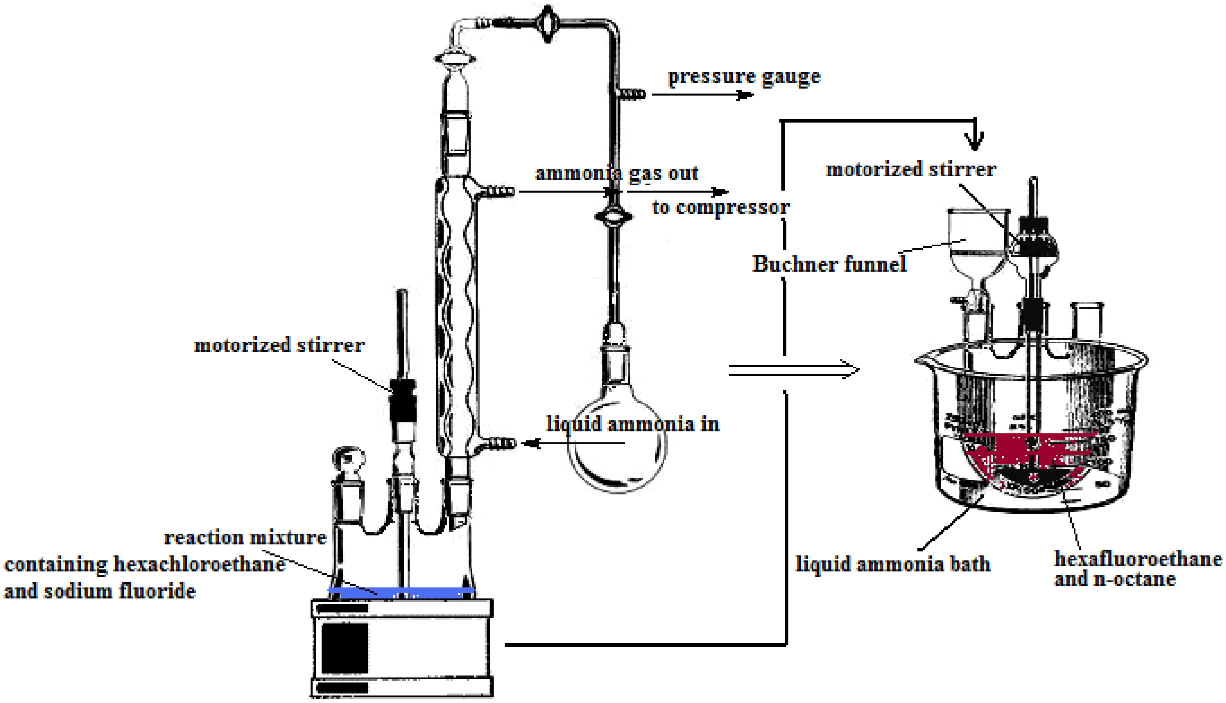
Into a special apparatus as illustrated below, purge the apparatus with dry argon gas for 5 minutes at a flow rate of 500 mg per/minute. Thereafter, turn on the heating mantle, and heat the reaction chamber to 150 Celsius. When the reaction chamber reaches 150 Celsius, turn on the UV light source and make sure it points directly to the center of the reaction chamber. Thereafter, begin to pass in at a flow rate of 50 mg per/minute, dry ethane gas, and immediately thereafter, begin to pass in dry chlorine gas at a flow rate of 750 mg per/minute while heating the reaction chamber to 150 Celsius under direct UV light contact. Continue to heat the reaction chamber to 150 Celsius until 109 grams of ethane gas, and 1,542 grams of chlorine gas has been added. Note: during the reaction, a plurality of mixed chlorinated ethanes will form and re-circulate into the reaction chamber, and over time, the desired product of hexachloroethane will condense on the wall of the reaction flask, and in the Snyder column. Note: the desired amount of ethane gas will be added far before the desired amount of chlorine is added. Once 109 grams of ethane has been added, close the stopcock at the ethane end, and continue to pass in chlorine at the desired flow rate. Once 1,542 grams of chlorine gas has been added, close the chlorine stopcock, but continue to heat the reaction chamber/reaction mixture at 150 Celsius under direct UV light for 4 hours. After 4 hours, turn off the heating mantle, but continue to direct UV light on the reaction mixture until the temperature of the reaction mixture reaches room temperature. Once the reaction chamber/reaction mixture has cooled to room temperature, disassemble the upper portion of the apparatus as illustrated in the right illustration shown below, and then flush the remaining apparatus with 2500 milliliters of dry hexane to dissolve out any desired product of hexachloroethane (that may be stuck to the walls of the apparatus) to form a solution of hexachloroethane/hexane. Thereafter, disassemble the apparatus, and then filter the hexane/hexachloroethane mixture to remove any insoluble impurities, using suction filtration, and then place this filtered hexane/hexachloroethane mixture into a rotary evaporator and evaporate-off the hexane under moderate vacuum and at a temperature of 50 Celsius until dry solid remains. When dry solid remains, stop the evaporation process, and allow the dry solid that remains to cool to room temperature. Once it has, recover the pasty mass of hexachloroethane, and save for step 2.



Setup for chlorination of ethane.

Step 2: Preparation of hexafluoroethane

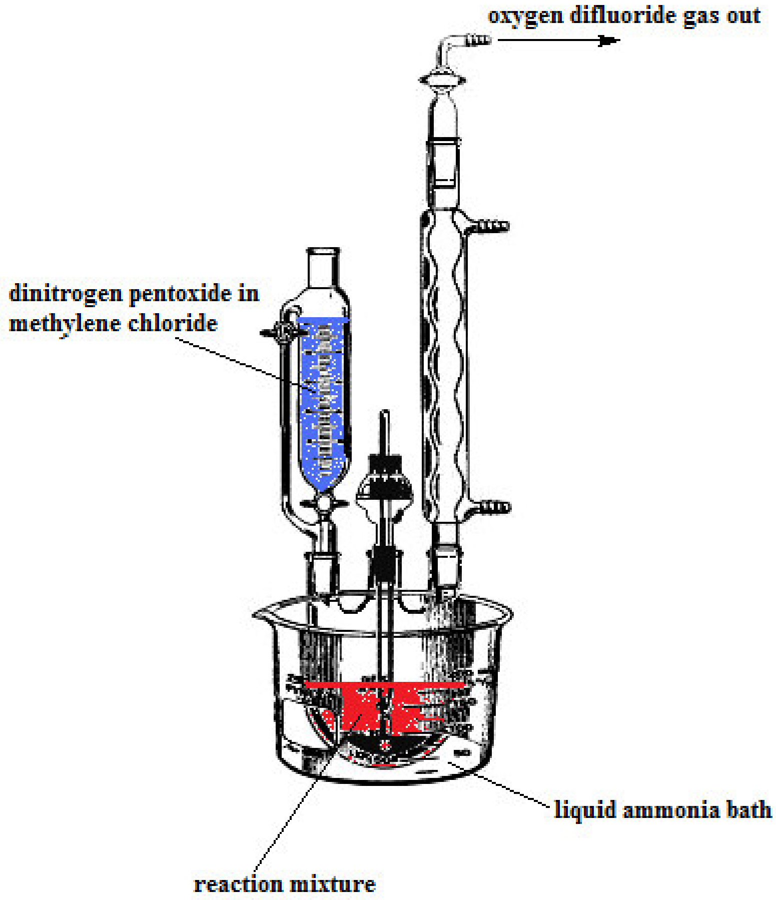
Into a another special apparatus as illustrated below, place into the 3-neck flask the product obtained from step 1, followed by 3000 milliliters of n-octane, followed by 923 grams of anhydrous sodium fluoride, and then assemble the apparatus, and begin to pump liquid ammonia into the condenser as illustrated. Thereafter, turn on the heating mantle and begin a reflux at 120 Celsius, while vigorously stirring the reaction mixture. Continue to reflux and rapidly stir the reaction mixture for 6 hours at 120 Celsius. After 6 hours, turn off the heating mantle and continue to stir the reaction mixture during the cool down process. Note: continue to pump in liquid ammonia into the condenser during the entire reaction, and cool down period to keep the desired product of hexafluoroethane from evaporating off, which is a gas at room temperature. Once the reaction mixture has cooled to room temperature, quickly disassemble the apparatus, and then filter this cooled reaction mixture using a Buchner funnel using suction filtration as shown in the right illustration shown below to remove the insoluble sodium chloride. Make sure to follow the right illustration using a liquid ammonia bath as shown. Once the reaction mixture has been suction filtered remove the Buchner funnel, and then save the 3-neck flask contained in the liquid ammonia bath illustrated in the right illustration as shown below for step 3.



Setup for fluorinating hexachloroethane, followed by filtration to remove insoluble impurities. Hexafluoroethane is a gas at room temperature and hence must be cooled. The right illustration is to be used in step 3.

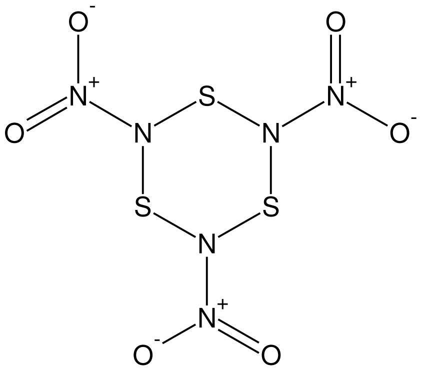
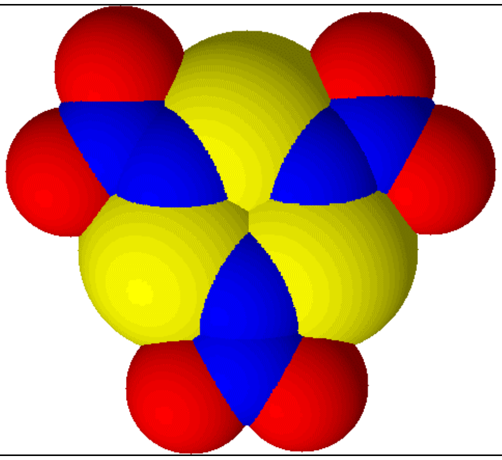
Step 3: Preparation of hexanitroethane

Into the apparatus as illustrated below and containing the hexafluoroethane and n-octane (obtained in step 2), place 5 grams of platinum sponge, and then assemble the apparatus and then pour into the addition funnel, 1/3rd of a nitrating mixture made by adding and dissolving 1,194 grams of dinitrogen pentoxide into 3000 milliliters of methylene chloride. Thereafter, begin a rapid drip of the dinitrogen pentoxide into the reaction mixture under rapid agitation while maintaining a low temperature utilizing the liquid ammonia bath. Note: more liquid ammonia will have to be added during the nitrating process as it will evaporate and hence, need to be replaced with fresh liquid ammonia. Once this 1/3rd portion of dinitrogen pentoxide in methylene chloride has been added, add in the next 1/3rd portion of dinitrogen pentoxide in methylene chloride into the addition funnel, and then begin a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while maintaining a liquid ammonia temperature under rapid agitation. Once this 1/3rd portion of dinitrogen pentoxide in methylene chloride has been added, add into the addition funnel the final 1/3rd portion of dinitrogen pentoxide in methylene chloride, and begin a slow drip of this dinitrogen pentoxide in methylene chloride into the reaction mixture while maintaining a liquid ammonia temperature under rapid stirring. Once all the dinitrogen pentoxide in methylene chloride has been added, remove the liquid ammonia bath, and then replace it with a heating mantle, and then reflux this entire reaction mixture at 80 Celsius for 6 hours. After 6 hours, stop the refluxing process, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Once it has, disassemble the apparatus, and pour the entire reaction mixture into a distillation apparatus (not shown) and distill-off the methylene chloride at 40 Celsius. Once no more methylene chloride passes over, stop the distillation process and then allow the remaining reaction mixture to cool to room temperature. Once it has cooled to room temperature, disassemble the distillation apparatus, and then pour the entire remaining mixture into a large beaker, and then add in all at once a solvent mixture prepared by mixing 1500 milliliters of diethyl ether into 1500 milliliters of acetone, and then stir this new mixture for 1 hour at room temperature. Note: once this solvent mixture is added, the desired product of hexanitroethane will precipitate. After stirring for 1 hour, filter-off the insoluble desired product of hexanitroethane using suction filtration, wash with three 1500-milliliter portions of hot water, and then suction dry as best as possible. Note: at this point you can pluck-out the platinum sponge. Finally, place the washed filtered dried desired product of hexanitroethane into an oven and bake at 105 Celsius for 48 hours. After 48 hours, turn off the oven, and then allow the desired product to cool to room temperature; thereafter, collect it, and then store it in an airtight container until use.


oxygen difluoride gas out
dinitrogen pentoxide in methylene chloride
liquid ammonia bath
reaction mixture

Setup for nitrating hexafluoroethane.

Theoretical Preparation 38: 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane; TNTTTA; Triazine; Compound T-2,4,6

 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $N_6O_6S_3$
Formula Weight = 276.2316
Composition = N(30.42%) O(34.75%) S(34.82%)
Molar Refractivity = 53.80 ± 0.4 cm^3
Molar Volume = 109.9 ± 5.0 cm^3
Parachor = 413.0 ± 6.0 cm^3

Index of Refraction = 1.968 ± 0.03
Surface Tension = 199.3 ± 5.0 dyne/cm
Density = 2.51 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 21.32 ± 0.5 10^{-24}cm^3
Monoisotopic Mass = 275.904142 Da
Nominal Mass = 276 Da
Average Mass = 276.2316 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7590 estimated using infrared spectroscopy	Flammable solid. Burns forming nitrogen oxides and sulfur dioxide. Mixtures with powdered magnesium explode when touched by a spark or flame. Keep out of contact with red phosphorus which forms a shock and friction sensitive composition.	Moderately stable to shock depending on the substance, i.e., forms shock sensitive mixtures with various reducing agents; when pure is mostly stable towards heat, shock, percussion, and friction. A secondary explosive. Requires a blasting cap or detonator for initiation.	Readily oxidized by strong oxidizing agents. Can be reduced with tin and hydrochloric acid. Keep out of contact with potassium permanganate and sulfuric acid—this mixture detonates spontaneously.	Light yellow-lime to yellowish crystalline monoclinic crystals, or yellowish white granules or powder. Melting point: 107 Celsius; Boiling point: decomposes when heated to 390 Celsius. Insoluble in water, and very very slowly hydrolyzed by water. Insoluble in all known solvents, but readily soluble in carbon disulfide and liquid ammonia

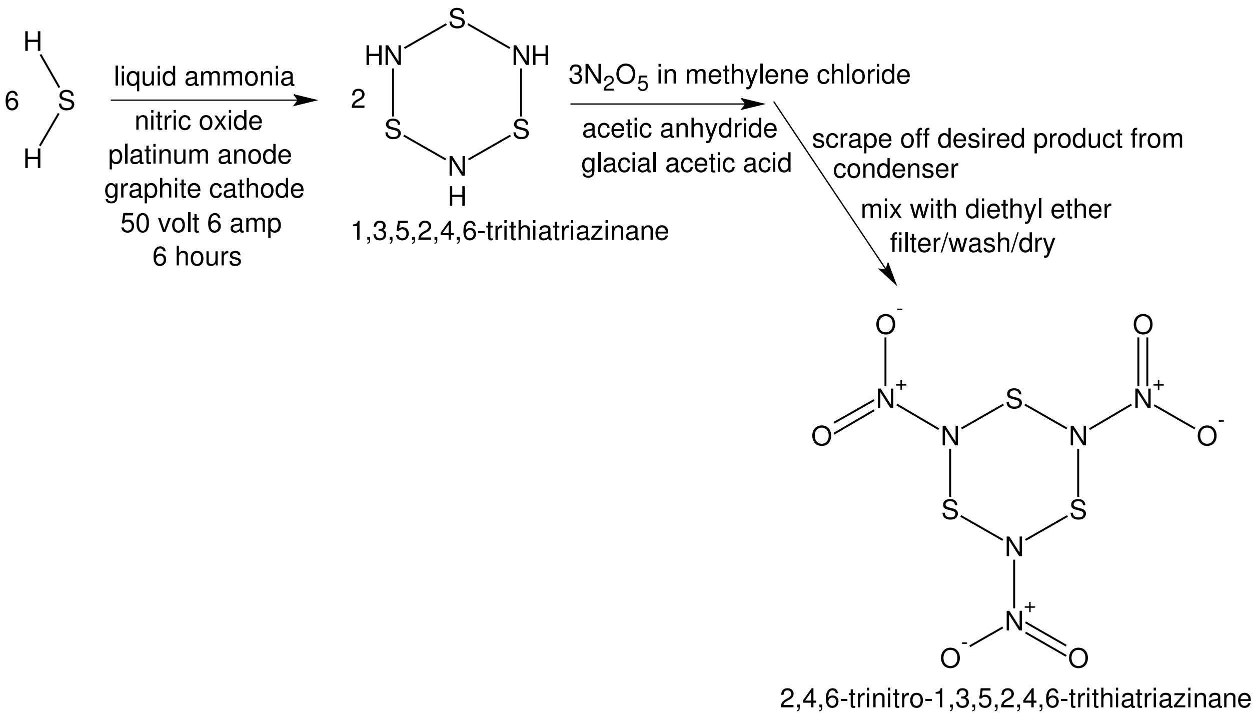
Estimated Compound summary: 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane is a fascinating cyclic nitro sulfur compound with a gas ratio of 100%. The explosive is readily melted and casted into molds for making shape charges, fillers for mortar shells, artillery shells, drop bombs, rocket warheads, and missile warheads. 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane when molten is insoluble in all known molten explosives, hence if it is to be melted down and used as an explosive component it can only be melted and used by itself. Because of its 100% gas ratio, it makes for super powerful shape charges that have 2 ½ times the penetrating power as Comp B; hence 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane demonstrates a remarkable use in making anti-tank weapons and anti-ship missiles due to its extreme penetrating power. 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane can either be dead pressed under 3,000 psi into tubes or melted down and casted into blocks for use in demolition operations for a variety of purposes—it can be granulated and sprayed into bore holes for blasting hard rock in outside demolition operations such as tree clearing or removing rock for road building, but it should not be used in mine shafts due to the toxic fumes it gives off after detonation. Other then its use as a high explosive, it demonstrates great potential for use in making pyrotechnic compositions. 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane can be used to make high performance rocket fuels when mixed with powdered aluminum and cured with epoxy, or it can be used in making high performance gun propellants when mixed with a high energy plasticizer and allowed to cure; however, this area of mixing 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane with reducing agents needs to be more carefully explored, as mentioned before it can form some shock sensitive substances with certain reducing agents mainly powdered magnesium and red phosphorus, and these mixtures can detonate very easily so caution must be taken. Nonetheless, 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane forms stable very powerful high performance gun propellants when mixed with a high energy plasticizer and a inert filler and allowed to cure—these gun propellants can be used in rifle casings, artillery propellants, propellants for high caliber shells such as battleship gun projectiles, and high velocity projectile propellants for tanks, but should not be used in mortar propellants as the mortar tube tends to explode upon ignition of the propellant. 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane shows potential use in making fireworks, but as noted, its mixtures with reducing agents has not been thoroughly investigated.

Safety Info: Use caution when handling liquid ammonia, which is very volatile and highly irritating to the eyes nose and throat, avoid inhalation and skin contact. Wear gloves when connecting the power clamps, and do not touch the electrodes during the electrolysis process. Use proper ventilation and avoid inhalation of hydrogen sulfide gas, which is toxic. Avoid inhalation of nitric oxide, which is also toxic. Wear gloves when handling acetic anhydride and glacial acetic acid, both of which are corrosive and can cause skin burns. Use care when handling dinitrogen pentoxide, which is a powerful nitrating agent, avoid contact with organic material. Use maximum ventilation when handling diethyl ether, which is highly flammable and volatile and forms explosive mixtures with air. Also, carryout a peroxide test and only use fresh diethyl ether to avoid and to check for peroxides, which can explode.

Materials:

1. 500 milliliters of liquid ammonia	6. 250 milliliters of acetic anhydride
2. One platinum anode	7. 350 milliliters of glacial acetic acid
3. One graphite anode	8. 235 grams of dinitrogen pentoxide
4. 137 grams of dry hydrogen sulfide gas	9. 365 grams of methylene chloride
5. 104 grams of nitric oxide gas	10. 2250 milliliters of dry diethyl ether

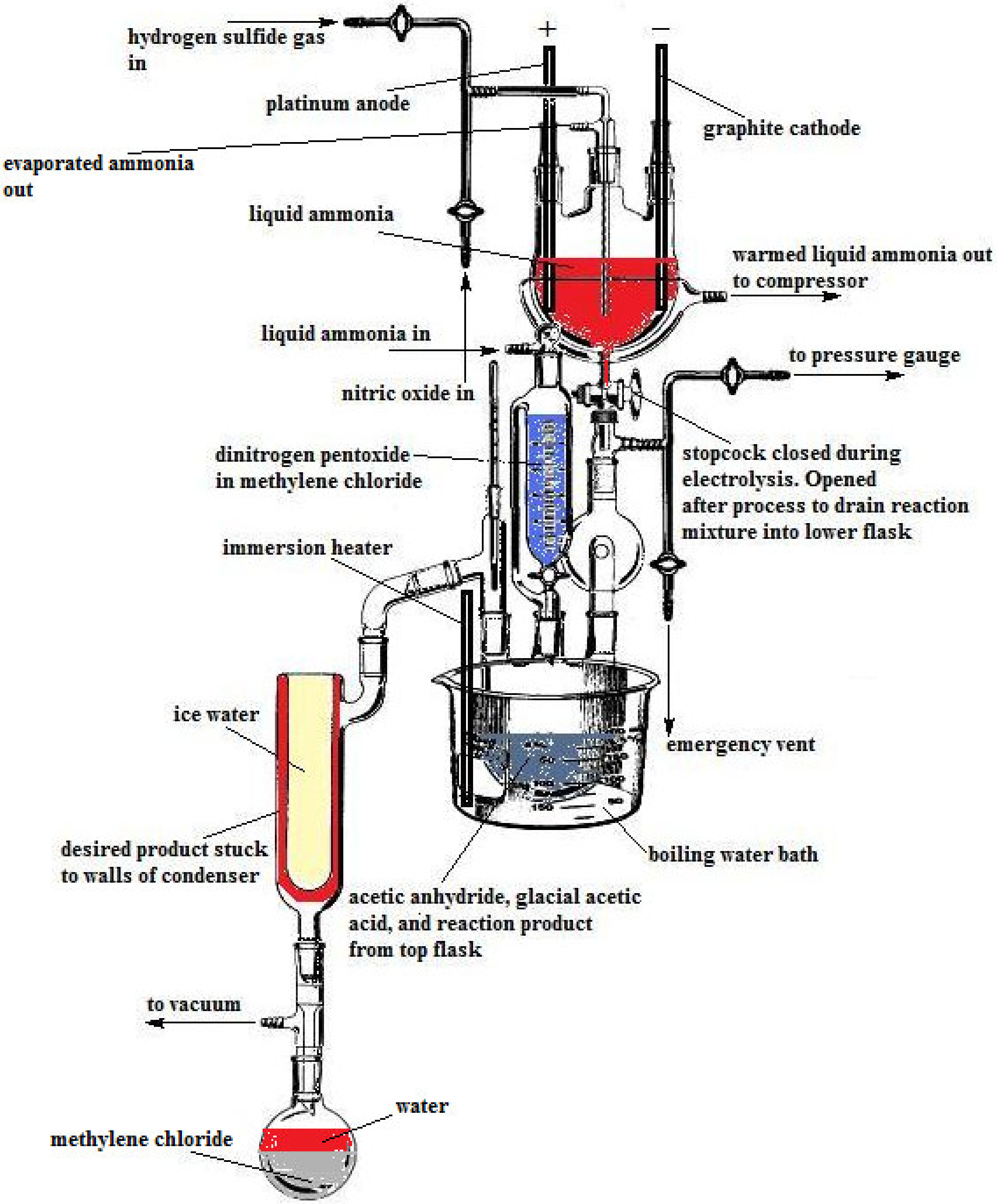
Reaction Equation:



Preparation:

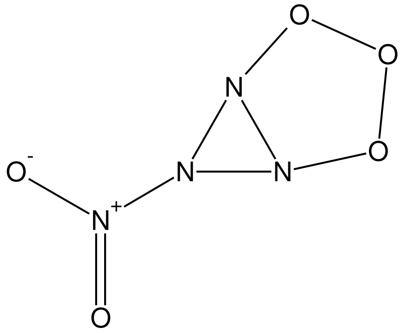
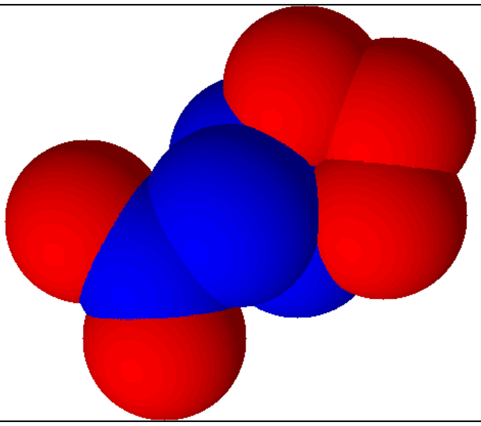
Step 1: One stop preparation of 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane

Into an advanced apparatus as illustrated below, place 500 milliliters of liquid ammonia into the upper 3-neck flask, and then insert the electrodes as illustrated. Thereafter, begin to pump liquid ammonia into the outer chamber to keep the liquid ammonia inside the 3-neck upper reaction chamber cooled to its proper temperature. Thereafter, begin to bubble into the liquid ammonia in the upper reaction flask 137 grams of dry hydrogen sulfide gas at a flow rate of 750 mg per/minute, and at the same time begin to bubble into the liquid ammonia reaction mixture 104 grams of nitric oxide gas at a flow rate of 300 mg per/minute. Immediately thereafter, secure the red clamp of the power supply to the platinum anode, and then secure the black clamp to the graphite cathode, and then turn on the power supply and electrolysize the liquid ammonia reaction mixture at 50 volt 6 amp for about 6 hours. Note: during this 6-hour period all the desired amount of hydrogen sulfide and nitric oxide would have been bubbled into the liquid ammonia reaction mixture. After 6 hours, turn off the power supply, and then disconnect the electrical clamps, and then stop pumping liquid ammonia into the outer chamber of the 3-neck flask and allow the reaction mixture to slowly warm but not to room temperature, meaning once the liquid ammonia coolant has stopped being pumped into the outer chamber, the liquid ammonia of the reaction mixture will begin to rapidly evaporate, so only allow this evaporation process to take place for about 30 minutes, and then open the bottom stopcock, and drain the remaining liquid ammonia reaction mixture into the lower 3-neck flask as illustrated below. Now, into this lower 3-neck reaction flask, quickly pour in 350 milliliters of glacial acetic acid, followed by 250 milliliters of acetic anhydride, and then assemble the apparatus as illustrated below using a dry ice condenser (that's what it is called but not always used with dry ice), and fill it with ice water. Immediately thereafter, insert an immersion heater into the water bath, and allow the water bath to begin to boil. Note: before this water bath begins to boil, pour into the addition funnel as illustrated 600 grams of a mixture of dinitrogen pentoxide in methylene chloride made by adding and dissolving 235 grams of dinitrogen pentoxide into 365 grams of methylene chloride, and then when the water in the water bath begins to boil, begin a moderate drip of the dinitrogen pentoxide/methylene chloride mixture from the addition funnel into the reaction mixture containing the glacial acetic acid and acetic anhydride. Once all the dinitrogen pentoxide/methylene chloride from the addition funnel has been added, close the stopcock to the addition funnel, and then allow the water bath to continue to boil for about 4 hours. Note: obviously during the reaction process and during the 4-hour period more water will have to be added to the water bath to replace evaporated water! Second note: During this 4-hour period, the desired product will condense on the walls of the dry ice condenser (containing ice water not dry ice). After 4 hours, remove the immersion heater, and allow the boiling water bath to cool to room temperature. Note: during the reaction process excess ammonia gas will be given off and will be carried away through the vacuum outlet so make sure you have a cold finger (not illustrated) to capture this gas so it does not enter the vacuum machine. Once the water bath has cooled to room temperature, disassemble the apparatus, and then take the dry ice condenser (containing ice water not dry ice in this case), and pour out the ice water, and then using a swindle or spatula, scrape off the desired product that will be stuck to the walls of this condenser, and place the scraped off product into a suitable sized beaker. Once the entire desired product has been scraped off the walls of the condenser and placed into a suitable sized beaker, pour into this beaker, 750 milliliters of dry diethyl ether, and then stir this mixture for 1 hour. After 1 hour, filter-off the insoluble desired product using suction filtration, and then wash with three 500-milliliter portions of fresh diethyl ether, and then suction dry as best as possible. Finally, take the filtered-off washed desired product and place it into a oven and then bake it at 50 Celsius for 24 hours. After 24 hours, turn off the oven and then allow the desired product to cool to room temperature before removing it. Once removing it, store it in an airtight container in a cool dry place until use.


hydrogen sulfide gas in
+
−
platinum anode
graphite cathode
evaporated ammonia out
liquid ammonia
warmed liquid ammonia out to compressor
liquid ammonia in
to pressure gauge
nitric oxide in
dinitrogen pentoxide in methylene chloride
stopcock closed during electrolysis. Opened after process to drain reaction mixture into lower flask
immersion heater
ice water
emergency vent
desired product stuck to walls of condenser
boiling water bath
acetic anhydride, glacial acetic acid, and reaction product from top flask
to vacuum
water
methylene chloride

Advanced setup for preparation of 2,4,6-trinitro-1,3,5,2,4,6-trithiatriazinane.

Theoretical Preparation 39: 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane; Trioxatriaza; NTTBH; Nitro trioxatriazabicyclo

 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = N_4O_5
Formula Weight = 136.0238
Composition = N(41.19%) O(58.81%)
Molar Refractivity = 20.86 ± 0.4 cm^3
Molar Volume = 42.9 ± 5.0 cm^3
Parachor = 169.6 ± 6.0 cm^3
Index of Refraction = 1.958 ± 0.03
Surface Tension = 243.6 ± 5.0 dyne/cm
Density = 3.16 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 8.27 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 135.986869 Da
Nominal Mass = 136 Da
Average Mass = 136.0238 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6185	Flammable when heated in air and ignited, burning forming nitrogen oxides.	Non-sensitive to shock, heat, percussion, pressure, and friction. Very stable. Will not detonate when struck with a sledgehammer or high-powered rifle bullet. Requires blasting cap or detonator for initiation. A secondary explosive.	Non-reactive. Very stable to chemical attack, but hydrolyzes when boiled with concentrated alkali. Explodes in contact with fluorine as expected.	Reddish to yellow monoclinic crystals. Pure compound is yellowish with a red tint; melting point 57 Celsius, Boiling point: decomposes when heated to 190 Celsius. Insoluble in water, insoluble in ether and acetone, hexane, and methylene chloride. Moderate

				solubility in liquid ammonia and liquid sulfur dioxide.

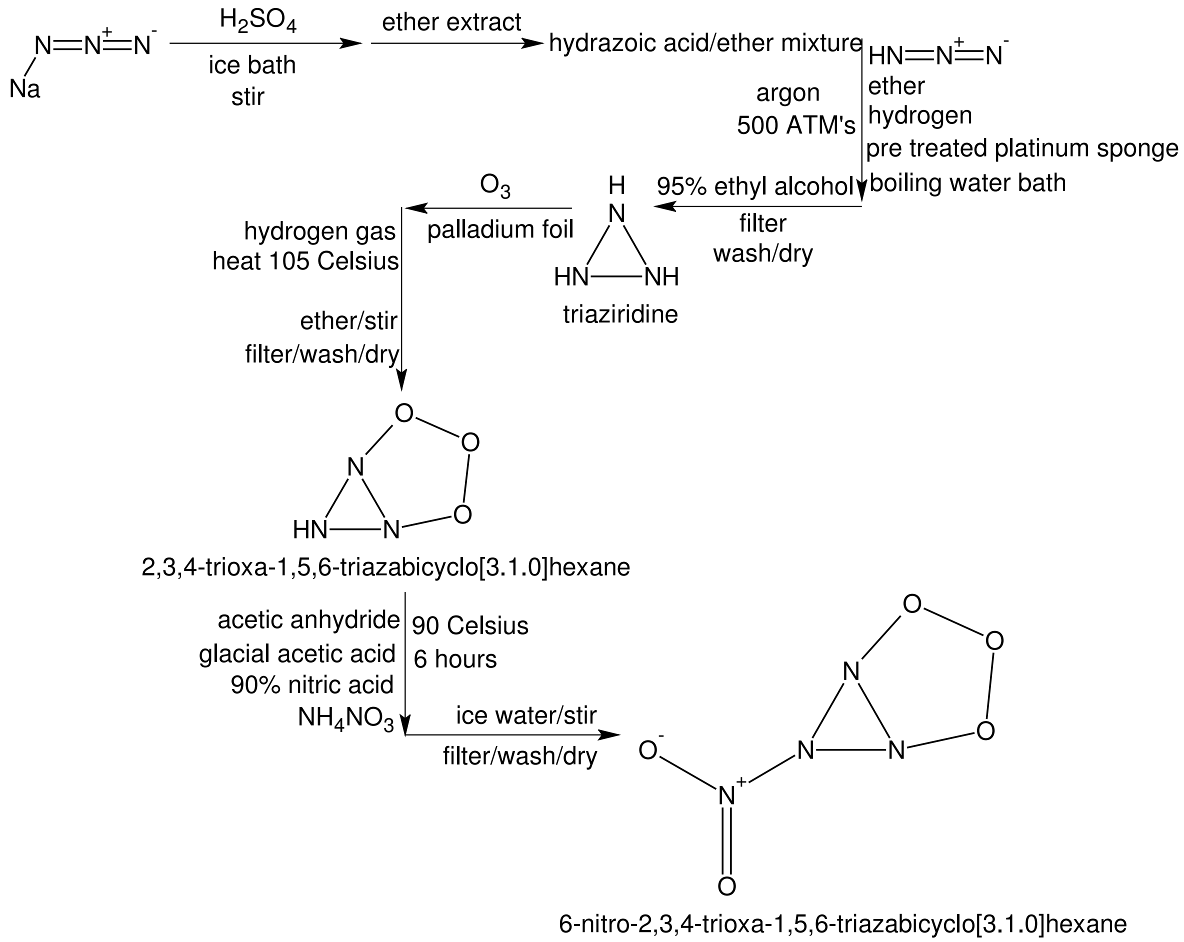
Estimated Compound summary: 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane is a cyclo trioxy nitramine with a low melting point, making it well suitable for melting and casting into molds for making shape charges, or pouring into mortar shells, artillery shells, or poured into other molds for making missile warheads and a wide variety of explosive munitions including land mines—if used to make land mines, the mines should only be used in place for no more then 5 years due to the explosives tendency to deteriorate over prolonged periods of time when exposed to the elements.. 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane tends to form a gel-like substance when melted and then ammonium nitrate added, and the mixture then allowed to cool. This gel-like material is very unique and can be used to form explosive munitions of odd shape and size; however, 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane is insoluble in most other high explosives such as TNT, RDX, PETN, HMX, ect. The stability of 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane is remarkable, and this makes for high interest in manufacturing explosives for various purposes mostly mortar shells, artillery shells, and high velocity tank fired projectiles, or high velocity explosive shells ranging from 20mm to 210mm. 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane as previously stated, when melted and ammonium nitrated added, a gel-like material is obtained; however if a high energy plasticizer is added, and material then cured under heat and pressure, a light weight rubber like flexible explosive is obtained. This rubber-like flexible material can be used in demolition operations when used in the form of sheets, or blocks. This rubber-like flexible material is very stable and chemically inert and water and solvent resistant. Note: either 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane used by itself, in the form of a gel with ammonium nitrate, or if used as a rubber-like flexible explosive should not be used in under ground mining operations due to toxic nitrogen oxide gasses that are produced upon detonation, but they can be used in demolition operation in open air situations such as demolishing buildings, destroying vehicles or equipment, or for clearing earth for road building. 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane shows no potential use for making high performance rocket propellants, high performance gun propellants, of in making pyrotechnic mixtures.

Safety Info: Wear gloves when handling concentrated sulfuric acid, and avoid contact with skin, clothing, and organic matter. Wear gloves when handling sodium azide, and avoid ingestion or inhalation of dust as sodium azide is very toxic. Extinguish all flames and sources of ignition when handling diethyl ether, which is highly volatile and forms explosive mixtures with air; also, avoid inhalation of diethyl ether, which is a narcotic in high concentrations. Use care when handling hydrogen, which forms explosive mixtures with air, remove all sources of ignition before using. Use caution when handling ozone gas, and avoid inhalation of ozone gas as it is toxic. Wear gloves when handling acetic anhydride and glacial acetic acid, both of which can cause skin irritation. Use great care when handling 90% nitric acid, which evolves toxic fumes of nitrogen oxides, use maximum ventilation.

Materials:

1. 150 grams of 98% sulfuric acid	8. 50 grams of palladium foil
2. 197 grams of sodium azide	9. 74 grams of dry ozone gas
3. 3000 milliliters of diethyl ether	10. 250 milliliters of acetic anhydride
4. 15 grams of platinum sponge	11. 300 milliliters of glacial acetic acid
5. Hydrogen gas from tank	12. 150 grams of ammonium nitrate
6. 7 grams of hydrogen gas	13. 165 grams of 90% nitric acid
7. 3450 milliliters of 95% ethyl alcohol	

Reaction Equation:



Preparation:

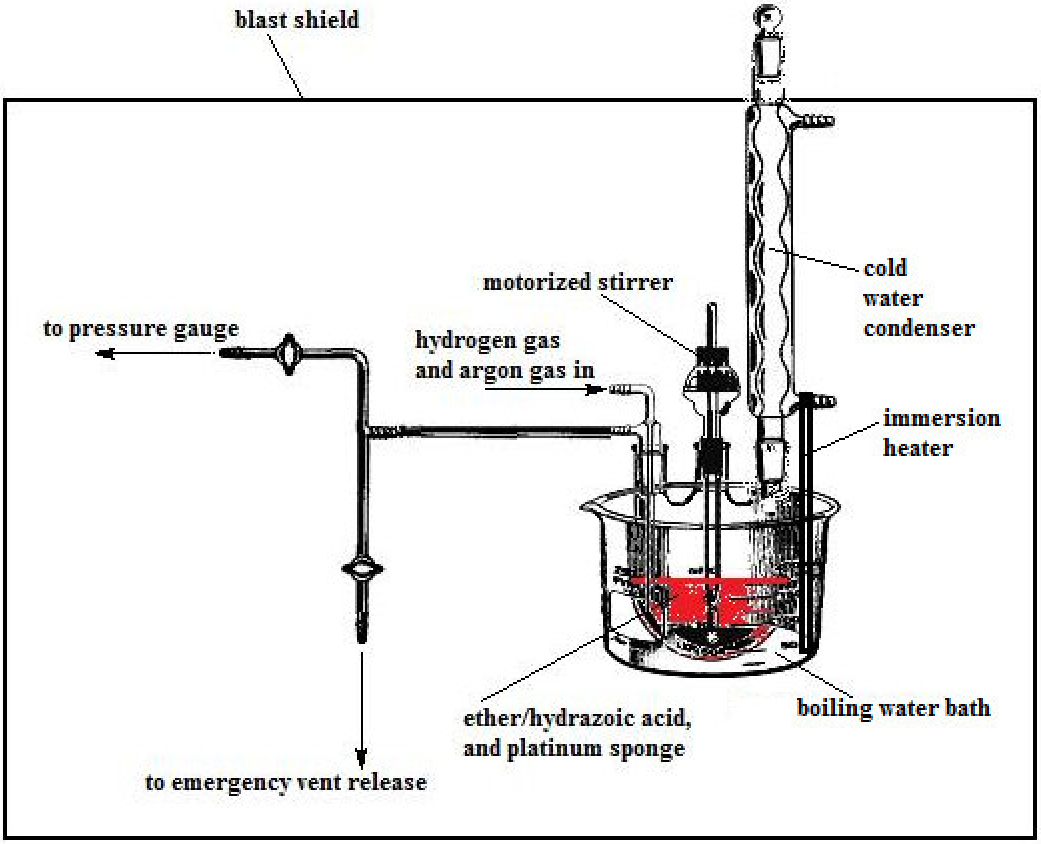
Step 1: Preparation of hydrazoic acid

Note: use a blast shield when carrying out this reaction as hydrazoic acid is highly explosive. Into a suitable sized beaker equipped with motorized stirrer and thermometer, place this beaker into an ice bath and chill the beaker to 0 Celsius. Once the beaker has chilled to 0 Celsius, pour in 150 grams of 98% sulfuric acid, and then chill this acid to 0 Celsius using the ice bath. Note: the ice will have to be replaced periodically during the chilling process. Once the concentrated sulfuric acid has reached a temperature of 0 Celsius, slowly add in, in small portions at time, 197 grams of sodium azide while vigorously stirring the acid mixture and maintaining its temperature around 0 Celsius at all times. After all the sodium azide has been added, continue to stir the acid reaction mixture at a temperature of 0 Celsius, under vigorous stirring for 1 hour. After 1 hour, stop the stirring process, and then extract this acid reaction mixture with three 500-milliliter portions of dry diethyl ether, and after the extraction, combine all three ether extracts, if not already done so, and then save this ether/hydrazoic acid mixture for step 2.

Step 2: Preparation of triaziridine

Preparing the platinum sponge: Into a suitable sized beaker, place into it 15 grams of platinum sponge, and then add in a small amount of diethyl ether. Thereafter, heat the platinum sponge to 500 Celsius, allowing the ether to rapidly boil away, and then pass dry hydrogen gas over the surface of the heated platinum sponge for 12 hours. Note: pass the hydrogen gas at a flow rate of 25 mg per/minute for 12 hours, while heating the platinum sponge to 500 Celsius. After 12 hours, stop passing hydrogen over the surface of the platinum sponge, and remove the heat source and allow the platinum sponge to cool to room temperature.
Preparation of triaziridine: Once the platinum sponge has cooled to room temperature quickly place it into a three neck flask and then pour into this 3-neck flask the diethyl ether/hydrazoic acid mixture prepared in step 1, and then assemble the apparatus as illustrated below. Now, turn on the immersion heater, and allow the water bath to boil, and then purge the

apparatus with dry argon gas, and then create a pressure of 500 ATM's. When the inside of the reaction apparatus has reached 500 ATM's, and the water bath begins to boil, begin to pass into this reaction mixture 7 grams of hydrogen gas at a flow rate of 150 mg per/minute while heating the reaction mixture and maintaining a vigorous stirring under a pressure of 500 ATM's. After the addition of the hydrogen gas, continue to heat the reaction mixture via the boiling water bath, and continue to stir the reaction vigorously for 4 hours under a argon purge at 500 ATM's. Note: during this 4-hour period, more water will have to be added to the water bath to replace evaporated water. After 4 hours, remove the argon purge, stop passing in hydrogen gas, and release the pressure gauge and bring the pressure inside the reaction apparatus to standard atmospheric pressure while continuing to rapidly stir the reaction mixture. Thereafter, remove the immersion heater, and allow the reaction mixture to cool to room temperature, and then pour the entire reaction mixture into a large beaker, and then pluck out the platinum sponge, followed by pouring into this beaker 1500 milliliters of 95% ethyl alcohol, and then stir this new mixture for 1 hour. After 1 hour, decant-off the upper liquid desired product of triaziridine or by using a separatory funnel, and save this liquid desired product of triaziridine for step 3.

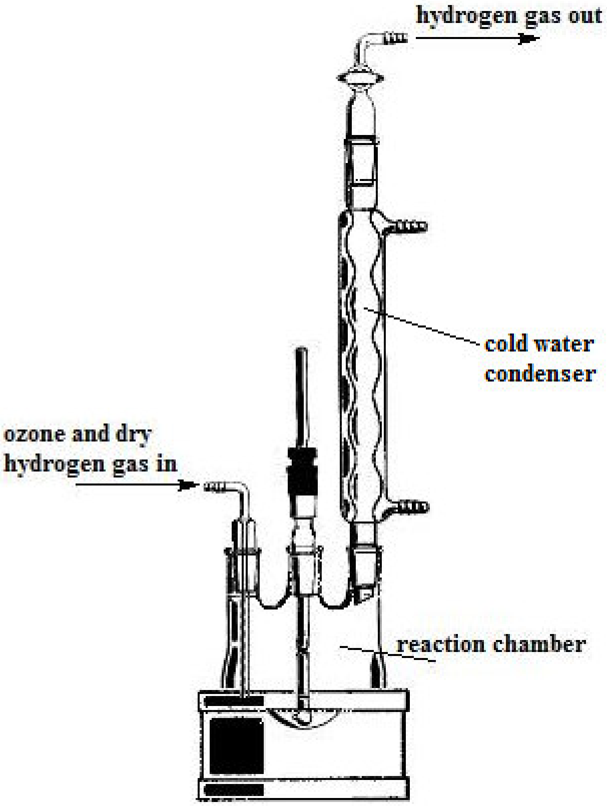


Setup for preparation of triaziridine.

Step 3: Preparation of 2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane

Into a apparatus as illustrated below, place the liquid product from step 2 into the 3-neck reaction flask followed by 50 grams of palladium foil, and then assemble the apparatus as illustrated below. Thereafter, turn on the heating mantle, and heat the 3-neck reaction flask to 105 Celsius, and when its temperature reaches 105 Celsius, begin to pass into the 3-neck reaction flask dry ozone gas at a flow rate of 50 mg per/minute, and at the same time, pass in dry hydrogen gas at a flow rate of 100 mg per/minute while heating the 3-neck reaction flask to 105 Celsius with stirring to circulate the gasses properly. Note: the hydrogen is merely a carrier, and more hydrogen gas will be evolved then that originally passed into the reaction flask. Continue to circulate the gasses in the 3-neck reaction flask while heating the reaction chamber to 105 Celsius until a total of 74 grams of dry ozone gas has been added. Note: the amount of hydrogen gas added is irrelevant; just continue to pass hydrogen gas into the reaction chamber at its proper flow rate as described. Once 74 grams of dry ozone gas has been added, stop the addition of ozone gas, and stop the passing of hydrogen gas, but continue to stir the contents of the reaction flask, while turning off the heating mantle and allow the contents in the reaction flask to cool to room temperature. Once the contents in the reaction flask have cooled to room temperature, remove the cold-water condenser and then pour in 1500 milliliters of dry diethyl ether, and then continue to stir the reaction mixture for 1 hour. After 1 hour, stop stirring and then filter-off the insoluble desired product of 2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane using suction filtration, and then wash with three

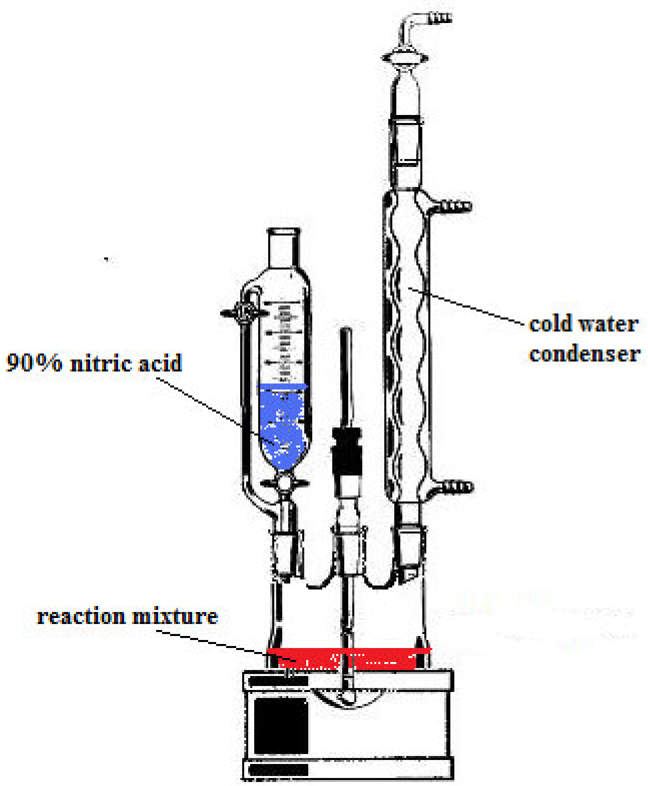
500-milliliter portions of hot water, and then suction dry as best as possible. Thereafter, keep this suction dried product of 2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane for step 4.



Setup for the preparation of the 2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane.

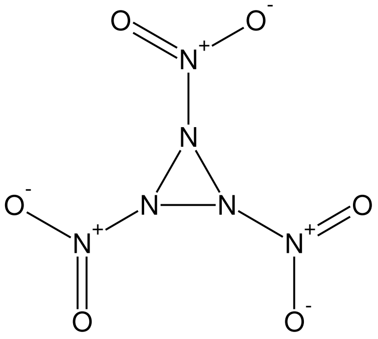
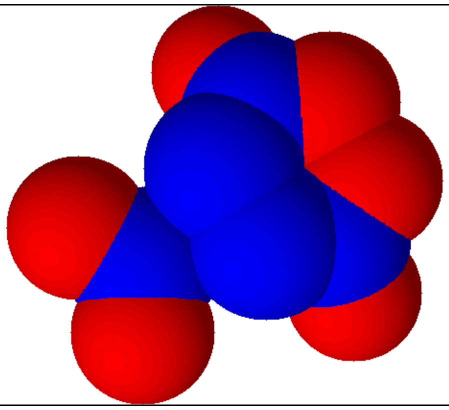
Step 4: Preparation of 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane

Into a suitable apparatus like the one illustrated below, place 250 milliliters of acetic anhydride, followed by 300 milliliters of glacial acetic acid, followed by 150 grams of ammonium nitrate, followed by the product obtained from step 3. Thereafter, turn on the heating mantle, and heat the reaction mixture to 90 Celsius under rapid agitation. Once the contents of the reaction mixture have reached 90 Celsius, pour in 165 grams of 90% nitric acid into the addition funnel, and begin a slow drip of this 90% nitric acid into the reaction mixture while heating the reaction mixture to 90 Celsius under rapid stirring. Once all of the 90% nitric acid has been added, continue to stir the reaction mixture and heat it at 90 Celsius for 6 hours. After 6 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature, but continue to rapidly stir the reaction mixture during this cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into 1500 milliliters of ice water, and then stir this new mixture for 2 hours. After 2 hours, filter-off the insoluble desired product of 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane using suction filtration, and then wash this filtered-off product with three 500-milliliter portions of 95% ethyl alcohol, and then suction dry as best as possible. Finally, take this suction dried product of 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane and place it onto a large shallow tray, and allow it to air-dry for 48 hours. After 48 hours, scrape up the desired product, and then store in any suitable container in a cool dry place until use.



Setup for nitration of 2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane.

Theoretical Preparation 40: trinitrotriaziridine; TNTA; Nitrotriaziridine

 trinitrotriaziridine	
Expanded structure	3D structure

Molecular data:

Molecular Formula = N_6O_6
Formula Weight = 180.0366
Composition = N(46.68%) O(53.32%)
Molar Refractivity = 29.75 ± 0.4 cm^3
Molar Volume = 67.7 ± 5.0 cm^3
Parachor = 255.8 ± 6.0 cm^3
Index of Refraction = 1.829 ± 0.03
Surface Tension = 202.7 ± 5.0 dyne/cm
Density = 2.65 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 11.79 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 179.987932 Da

Nominal Mass = 180 Da
Average Mass = 180.0366 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
8090; estimated using infrared spectroscopy.	Flammable; burns very slowly with a colorless flame.	Non-sensitive to shock, percussion, heat, pressure, or friction. Very stable will detonate when struck very hard with a hammer or when struck by a rifle bullet. A secondary explosive. Requires a blasting cap or detonator for initiation.	Non-reactive; very stable. Cannot be reduced using the usual chemical techniques. Virtually chemically inert.	Snow white crystalline mass, white powder or granules. Melting point: 67 Celsius; boiling point: 213 Celsius. Insoluble in water, and most organic solvents. Moderate solubility in methylene chloride. Slight solubility in acetic anhydride and liquid sulfur dioxide. Moderate solubility in carbon disulfide.

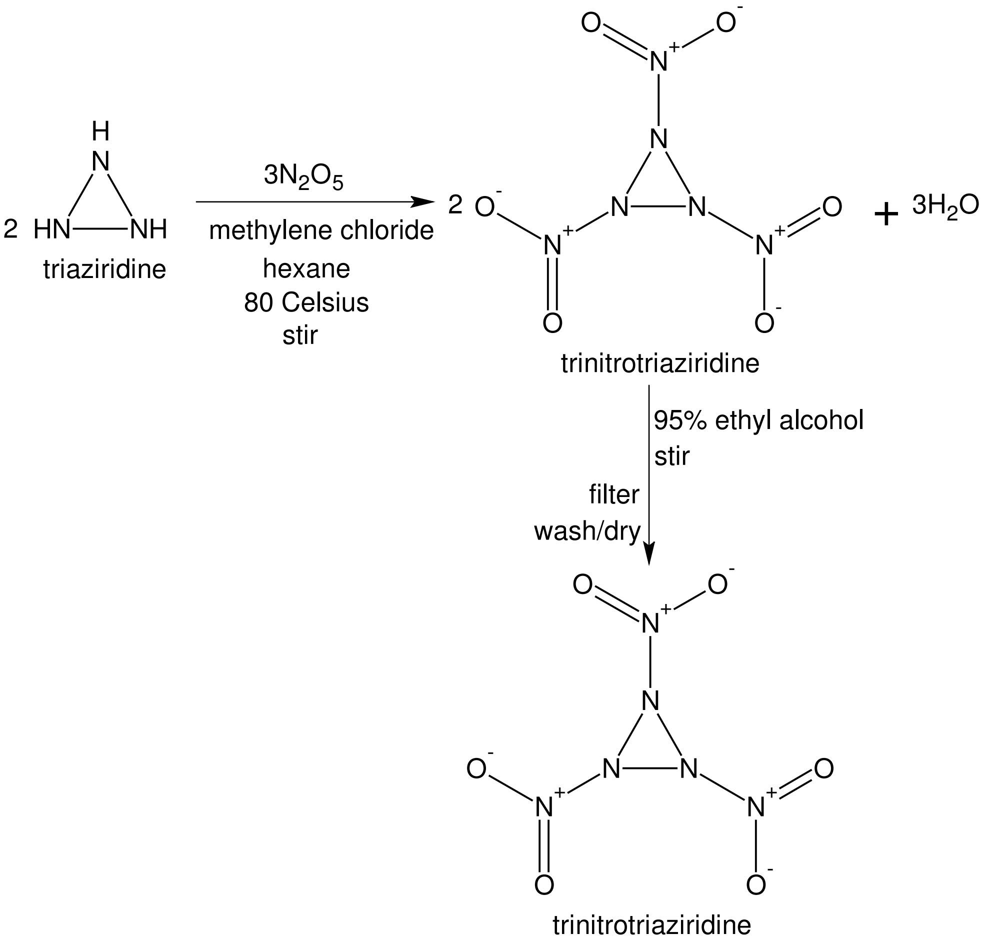
Estimated Compound summary: trinitrotriaziridine is the god father of 6-nitro-2,3,4-trioxa-1,5,6-triazabicyclo[3.1.0]hexane, and it has the perfect physical properties to become a fascinating new explosive for military and commercial use. Trinitrotriaziridine is a high explosive fitting into the family of cyclic nitramines and trinitrotriaziridine could be considered the child of the high explosive RDX. Trinitrotriaziridine has remarkable stability with a relatively low and very useful melting point allowing for it to be melted down and cast into molds for making shape charges of all kinds for military operations, and for making linear shape charges of various sizes for military and commercial demolition operations. Trinitrotriaziridine shows amazing potential for mass production in replacing older and ageing explosives for making mortar shells, artillery shells, drop bombs, torpedoes, land mines, anti-tank weapons, and high velocity projectiles. Trinitrotriaziridine when molten is soluble in molten RDX, and HMX, but insoluble in most other molten explosives; therefore trinitrotriaziridine can be mixed with RDX and HMX for making very stable long-term explosive compositions for many different operations. Although rarely explored, and warranted for further investigation is the mass manufacturing of high performance gun propellants using trinitrotriaziridine as a main component due to its 100% gas ratio upon ignition. Mixtures of trinitrotriaziridine with powered metals or reducing agents have yet to be explored but are warranted for further investigation.

Safety Info: Extinguish all flames and sources of ignition before using hexane, which is highly flammable. Use caution when handling dinitrogen pentoxide, which is very volatile and corrosive.

Materials:

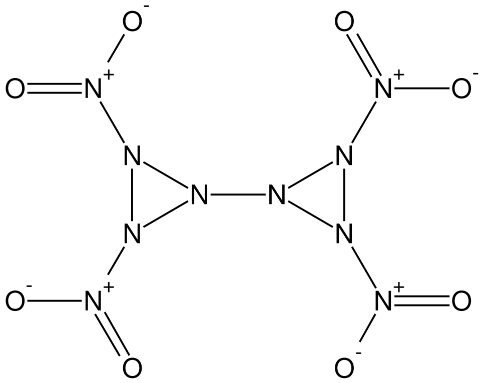
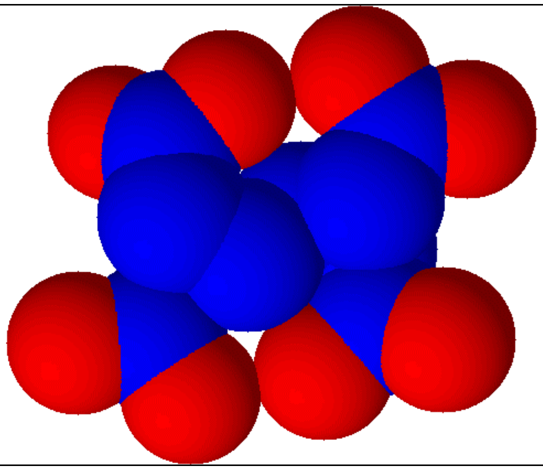
1. 200 grams of triaziridine (prepared in theoretical preparation 39, step 2)	4. 2000 milliliters of methylene chloride
2. 300 milliliters of dry hexane	5. 7150 milliliters of 95% ethyl alcohol
3. 720 grams of dinitrogen pentoxide	

Reaction Equation:



Preparation: Into a large 3-neck flask sitting inside a round bottom heating mantle, and equipped with cold-water condenser, thermometer, and motorized stirrer, pour in 200 grams of triaziridine (prepared in theoretical preparation 39 step 2), followed by 300 milliliters of dry hexane. Then begin to stir this mixture, and then turn on the heating mantle and bring the temperature of the contents in the 3-neck flask to around 80 Celsius under reflux. Immediately thereafter pour into the addition funnel 1/3rd of a mixture obtained by dissolving 720 grams of dinitrogen pentoxide into 2000 milliliters of methylene chloride. Once the contents in the 3-neck flask begins to boil under reflux, begin a moderate drip of the 1/3rd portion of dinitrogen pentoxide/methylene chloride mixture from the addition funnel while vigorously stirring the reaction mixture and maintaining its temperature around 80 Celsius. Once all this 1/3rd portion of dinitrogen pentoxide/methylene chloride mixture has been added to the reaction mixture from the addition funnel, pour in another 1/3rd of the dinitrogen pentoxide methylene chloride mixture into the addition funnel, and then continue a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while vigorously stirring the reaction and maintaining its temperature under reflux at 80 Celsius. Once all this dinitrogen pentoxide/methylene chloride has been added to the reaction mixture, pour in the final 1/3rd of the dinitrogen pentoxide/methylene chloride mixture into the addition funnel, and then continue a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while vigorously stirring the reaction mixture and maintaining its temperature under reflux at 80 Celsius. Once all the dinitrogen pentoxide/methylene chloride has been added, continue to vigorously stir the reaction mixture and maintain a reflux at 80 Celsius for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during this cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a super large beaker, and then pour in 2500 milliliters of 95% ethyl alcohol, and then stir this new mixture for 1 hour at room temperature. Note: upon the addition of the 95% ethyl alcohol, all the desired product of trinitrotriaziridine will precipitate. After stirring this new mixture for 1 hour, filter-off the insoluble desired product using suction filtration, and then wash this filtered-off desired product with three 1500-milliters portions of fresh 95% ethyl alcohol, and then wash with three 1500-milliliter portions of cold water, and then suction dry as best as possible. Finally, take this suction dried desired product and place it onto a shallow pan or tray and allow it to air dry for 48 hours. After 48 hours the desired product should be scraped off the pan or tray and placed into an airtight container and stored in a cool dry place until use.

Theoretical Preparation 41: 2,2',3,3'-tetranitro-1,1'-bitriaziridine; Bitriaziridine; TNBTA

 2,2',3,3'-tetranitro-1,1'-bitriaziridine	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $N_{10}O_8$
Formula Weight = 268.0622
Composition = N(52.25%) O(47.75%)
Molar Refractivity = 45.40 ± 0.4 cm^3
Molar Volume = 80.7 ± 5.0 cm^3
Parachor = 364.1 ± 6.0 cm^3
Index of Refraction = 2.202 ± 0.03
Surface Tension = 413.5 ± 5.0 dyne/cm
Density = 3.31 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 17.99 ± 0.5 10^{-24}cm^3
Monoisotopic Mass = 267.990057 Da
Nominal Mass = 268 Da
Average Mass = 268.0622 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
8190; estimated using Molar volume and mass spectroscopy	Non-Flammable under normal conditions; will burn when heated to above 120 Celsius burning very slowly with a luminescent flame emitting toxic fumes of nitrogen oxides.	Very stable. Not sensitive to heat, shock, percussion, pressure, or friction. A secondary explosive. Requires a blasting cap or detonator for initiation.	Very chemically inert. Does not react with reducing agents or oxidizing agents under normal conditions. May explode if heated with potassium permanganate, or if heated with alkali metals, but stable at ordinary temperatures.	Light reddish crystalline solid, granules, or powder. Melting point: 89 Celsius; Boiling point: Begins to slowly decomposes when heated to above 275 Celsius, and rapidly decomposes when heated to 350 Celsius. Insoluble

				in water, and most organic solvents. Slightly soluble in methylene chloride, liquid ammonia, and carbon disulfide.

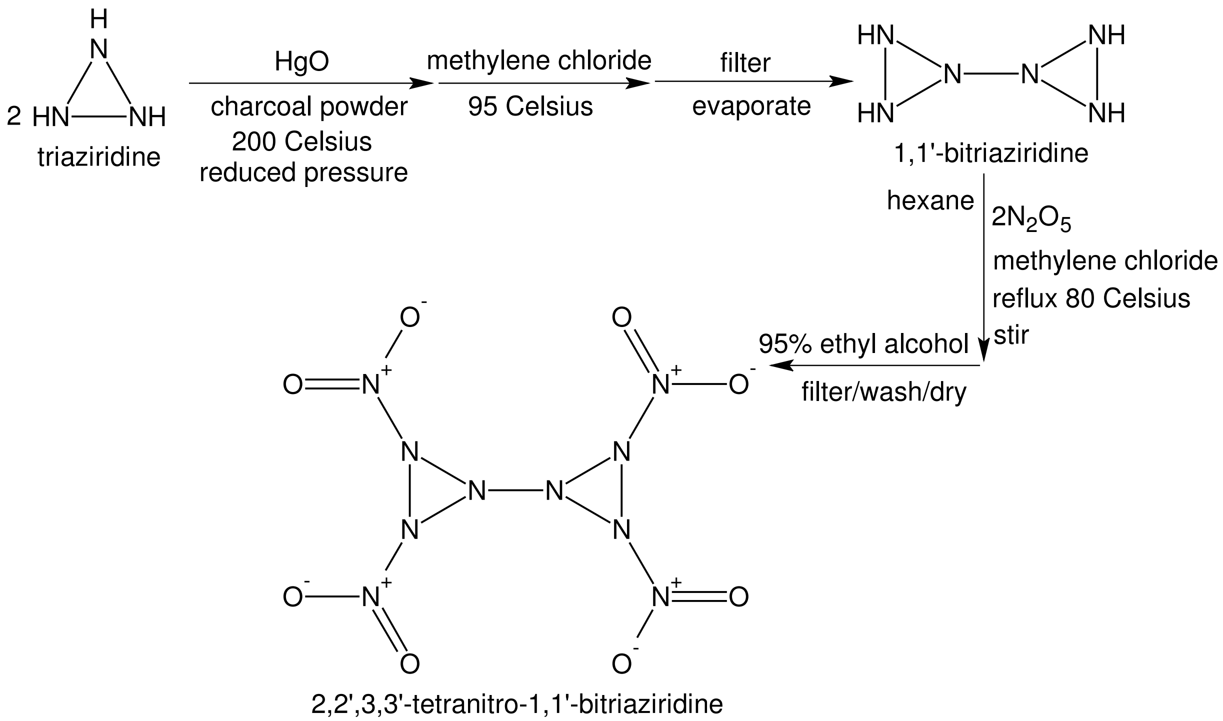
Estimated Compound summary: 2,2',3,3'-tetranitro-1,1'-bitriaziridine is another cyclic nitramine explosive of the RDX family with a dicyclic ring instead of a single one. 2,2',3,3'-tetranitro-1,1'-bitriaziridine is an amazing and outstanding high explosive with tremendous shattering power making it very useful for making penetrating explosive munitions that would be used to penetrate earth and ware structures, stone, concrete, sand bags, ect., ect., 2,2',3,3'-tetranitro-1,1'-bitriaziridine is the higher up brother of trinitrotriaziridine and is a fascinating new explosive that has not been thoroughly investigated. It has amazing physical properties in that it is very stable to chemical attack, and is relatively stable under temperatures of 270 Celsius. It can be easily melted and cast into molds of various sizes and shapes for making a wide variety of explosive charges for many different military and commercial operations. 2,2',3,3'-tetranitro-1,1'-bitriaziridine is more powerful of an explosive then its younger brother trinitrotriaziridine, and it can be either dead pressed into shells or bomb casings under 1500 psi, or it can be melted down and poured into shells and bomb casings and then allowed to cool. Molten 2,2',3,3'-tetranitro-1,1'-bitriaziridine is soluble in molten trinitrotriaziridine to form a unique explosive with a melting point of 91 Celsius, and forming a high explosive that is very resistant to the elements and can be used to make very powerful land mines, torpedoes, and demolition charges for use in wet weather environments; however molten 2,2',3,3'-tetranitro-1,1'-bitriaziridine is insoluble in most other molten explosives, but is soluble in molten RDX forming yet another unique explosive composition with similar effects and properties as with the trinitrotriaziridine/2,2',3,3'-tetranitro-1,1'-bitriaziridine composition. Other then that, 2,2',3,3'-tetranitro-1,1'-bitriaziridine is most likely to be used by itself for use in mortar shells, artillery shells, shape charges for anti-tank and demolition operations, land mines, demolition blocks for demo operations, missile warheads, torpedoes, and high velocity explosive munitions of all shapes and sizes. 2,2',3,3'-tetranitro-1,1'-bitriaziridine has little or no use for making pyrotechnic compositions, but this area of expertise has yet to be thoroughly investigated, but is doubtful that 2,2',3,3'-tetranitro-1,1'-bitriaziridine shows any remarkable use for making pyrotechnic compositions. Also, 2,2',3,3'-tetranitro-1,1'-bitriaziridine has little or no use for making high velocity gun propellants, or high performance rocket fuels, but as with potential pyrotechnic compositions the use of 2,2',3,3'-tetranitro-1,1'-bitriaziridine in making high performance gun propellants and/or high performance rocket fuels has yet to be thoroughly investigated but is doubtful of any potential use.

Safety Info: Wear gloves and avoid skin contact and ingestion of mercury oxide, which is highly toxic. Extinguish all flames before using hexane, which is highly flammable. 95% ethyl alcohol is also flammable but not volatile like hexane.

Materials:

1. 157 grams of triaziridine (prepared in theoretical preparation 39, step 2)	5. 750 milliliters of hexane
2. 2400 milliliters of methylene chloride	6. 378 grams of dinitrogen pentoxide
3. 378 grams of mercury-II-oxide	7. 3000 milliliters of 95% ethyl alcohol
4. 400 grams of charcoal powder	

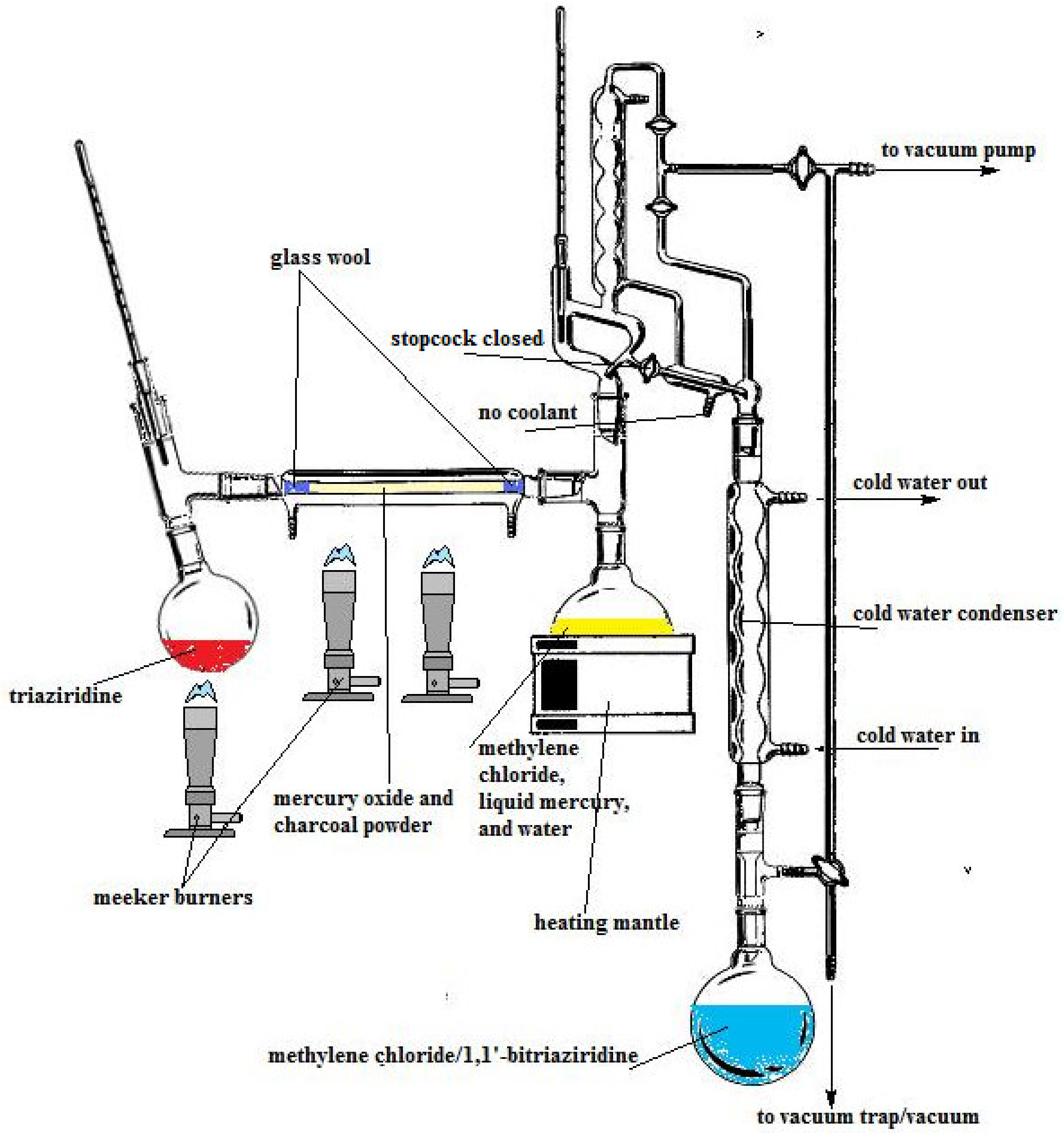
Reaction Equation:



Preparation:

Step 1: Preparation of 1,1'-bitriaziridine

Into an advanced apparatus as illustrated below, place 157 grams of triaziridine (prepared in theoretical preparation 39 step 2) into the proper flask as shown in the following illustration, and then place 900 milliliters of methylene chloride into the center round bottom flask as shown in the following illustration, and then turn on its heating mantle to 95 Celsius. Thereafter, into the condenser as shown, place glass wool in one end, and then add in 378 grams of mercury-II-oxide, followed by 400 grams of charcoal powder and then seal the other end of this condenser with glass wool, and then assemble this condenser back into the apparatus as shown. Now, turn on the vacuum pump and reduce the pressure inside the apparatus to 150 millimeters of mercury. Now using a meeker burner heat the triaziridine to 120 Celsius, and then place two additional meeker burners underneath the condenser containing the mercury oxide and charcoal powder and then ignite these meeker burners and heat the mercury oxide and charcoal powder at 200 Celsius. Note: during the process, the triaziridine will pass through the heated condenser containing the mercury oxide and charcoal forming 1,1'-bitriaziridine, which will eminently mix with the boiling hot methylene chloride and dissolve, and will be carried over into the lower receiving flask as shown in the bottom illustration. Continue to carryout the reaction utilizing the special apparatus until all the triaziridine has been boiled (reacted) away. Once all the triaziridine has evaporated and been reacted, remove the vacuum source, and allow the pressure inside the apparatus to reach standard atmospheric pressure, at the same time, extinguish all meeker burners (turn off the gas), and then allow the heated portions of the following illustration to cool to room temperature. Then, disassemble the apparatus and then take the single neck round bottom flask containing the methylene chloride/1,1'-bitriaziridine, and then quickly filter it using suction filtration, and then take this suction filtered liquid of methylene chloride/1,1'-bitriaziridine, and place it into a rotary evaporator, and evaporate-off the methylene chloride under mild vacuum and at a temperature of 39 Celsius until no more methylene chloride passes over. Once no more methylene chloride passes over, and a messy mass is left in the rotary evaporator, turn off the rotary evaporator, and then allow the left over remaining residue mass of desired product of 1,1'-bitriaziridine to cool to room temperature. Once it has cooled to room temperature, recover it, and save it for step 2.

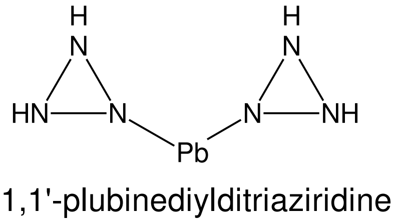
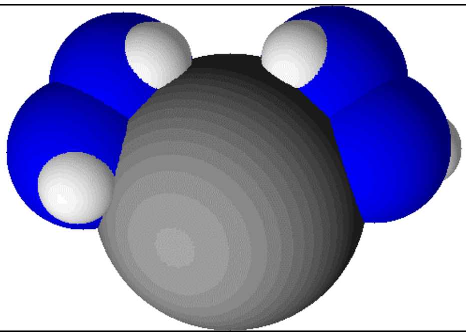


Setup for preparing 1,1'-bitriaziridine.

Step 2: Preparation of 2,2',3,3'-tetranitro-1,1'-bitriaziridine

Into a large 3-neck flask sitting inside a round bottom heating mantle, and equipped with cold-water condenser, addition funnel, thermometer, and motorized stirrer, pour in 750 milliliters of hexane, and then add in the messy mass of 1,1'-bitriaziridine obtained in step 1. Then begin to stir this mixture, and then turn on the heating mantle and bring the temperature of the contents in the 3-neck flask to around 80 Celsius under reflux. Immediately thereafter pour into the addition funnel 1/3rd of a mixture obtained by dissolving 378 grams of dinitrogen pentoxide into 1500 milliliters of methylene chloride. Once the contents in the 3-neck flask begins to boil under reflux, being a moderate drip of the 1/3rd portion of dinitrogen pentoxide/methylene chloride mixture from the addition funnel while vigorously stirring the reaction mixture and maintaining its temperature around 80 Celsius. Once all this 1/3rd portion of dinitrogen pentoxide/methylene chloride mixture has been added to the reaction mixture from the addition funnel, pour in another 1/3rd of the dinitrogen pentoxide methylene chloride mixture into the addition funnel, and then continue a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while vigorously stirring the reaction mixture and maintaining its temperature under reflux at 80 Celsius. Once all this dinitrogen pentoxide/methylene chloride has been added to the reaction mixture, pour in the final 1/3rd of the dinitrogen pentoxide/methylene chloride mixture into the addition funnel, and then continue a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while vigorously stirring the reaction mixture and maintaining its temperature under reflux at 80 Celsius. Once all the dinitrogen pentoxide/methylene chloride has been added, continue to vigorously stir the reaction mixture and maintain a reflux at 80 Celsius for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during this cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a large beaker, and then pour in 1500 milliliters of 95% ethyl alcohol, and then stir this new mixture for 1 hour at room temperature. Note: upon the addition of the 95% ethyl alcohol, all the desired product of 2,2',3,3'-tetranitro-1,1'-bitriaziridine will precipitate. After stirring this new mixture for 1 hour, filter-off the insoluble desired product using suction filtration, and then wash this filtered-off desired product with three 500-milliter portions of fresh 95% ethyl alcohol, and then wash with three 750-milliliter portions of warm water, and then suction dry as best as possible. Finally, take this suction dried desired product and place it onto a shallow pan or tray and allow it to air dry for 48 hours. After 48 hours the desired product should be scraped off the pan or tray and placed into an airtight container and stored in a cool dry place until use.

Theoretical Preparation 42: 1,1'-plubinediylditriaziridine; Lead diylditriaziridine; triaziridine lead nitride; Lead-DDTA

 H N HN N Pb N NH H N 1,1'-plubinediylditriaziridine	
Expanded structure	3D structure

Molecular data:

Molecular Formula = H_4N_6Pb
Formula Weight = 295.27196
Composition = H(1.37%) N(28.46%) Pb(70.17%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = Not available
Nominal Mass = Not available
Average Mass = Not available

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
4670	Non-Flammable. Explodes when rapidly heated or touched by a spark or flame.	Moderate sensitivity to shock and percussion, but stable towards heat, pressure and friction. A small	Generally non-reactive. Is slowly hydrolyzed by strong acids.	Reddish powder. Melting point: explodes; Boiling point: explodes. Explodes when

		sample explodes when heated above 200 Celsius, struck by a hammer, or touched by a spark or flame. A primary explosive.		heated to above 200 Celsius. Insoluble in all known solvents, including liquid ammonia and liquid sulfur dioxide.

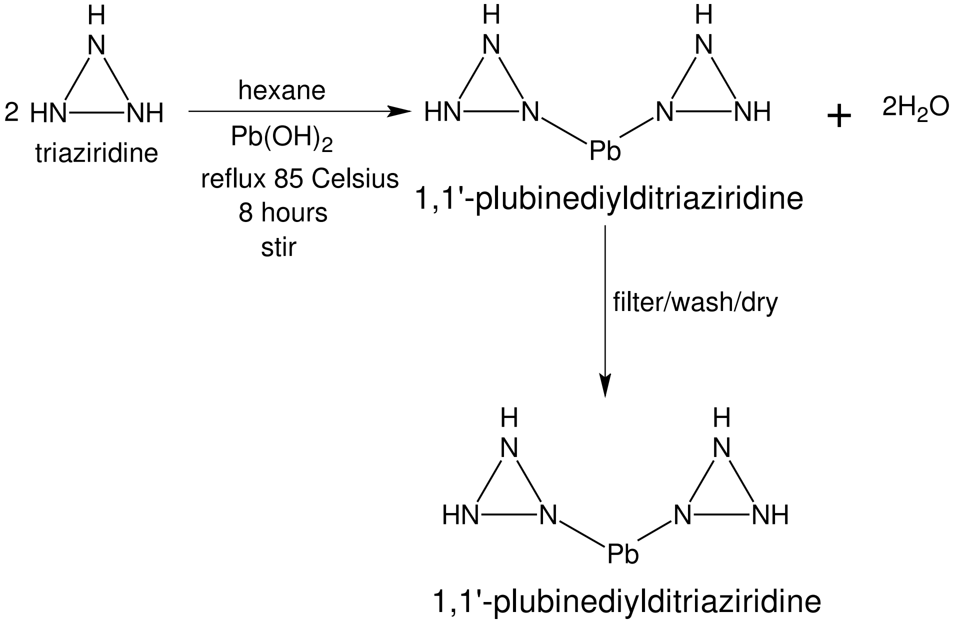
Estimated Compound summary: 1,1'-plubinediylditriaziridine is an interesting primary explosive that is designed to be an alternative to lead azide in blasting caps and detonators. It has remarkable stability (greater then lead azide), and does not have to be diluted for safety precautions. Even though a small sample explodes when struck with a hammer, it’s stability disallows it to be used in bullet primers; however, 1,1'-plubinediylditriaziridine can be used in electrical primers as seen in tank shells. Because of its stability, it is preferred over lead azide, which is less stable and has to be diluted with dextrose to stabilize it. 1,1'-plubinediylditriaziridine is a perfect primer for mortar shells, tank fired shells, artillery primers, and for use in impact fuses for mortar and artillery shells. 1,1'-plubinediylditriaziridine has remarkable properties and is sure to surpass the ageing lead azide, and other primary explosives for use in blasting caps, detonators, and point impact-point initiating impact fuses for use in tank fired projectiles, mortars shells, artillery, and any desired shell of any diameter and weight. Because of its stability, 1,1'-plubinediylditriaziridine can be used by itself in easy to ignite detonating cord that can be used in priming multiple demolition blocks for commercial and military demolition operations. This type of detonating cord is designed to replace the more common PETN detonating cord which has to be primed, unlike with 1,1'-plubinediylditriaziridine, which can be detonated using a flame, spark, or by electrical means. 1,1'-plubinediylditriaziridine can be used as a bursting charge in aerial fireworks but due to its difficulty and expense of manufacturing triaziridine the explosive will hardly replace the much cheaper black powder and chlorate and perchlorate pyrotechnic mixtures for use in fireworks; however pyrotechnic compositions containing 1,1'-plubinediylditriaziridine does warrant further investigation.

Safety Info: Extinguish all flames and sources of ignition when handling hexane, which is highly flammable. Wear gloves when handling Lead compounds.

Materials:

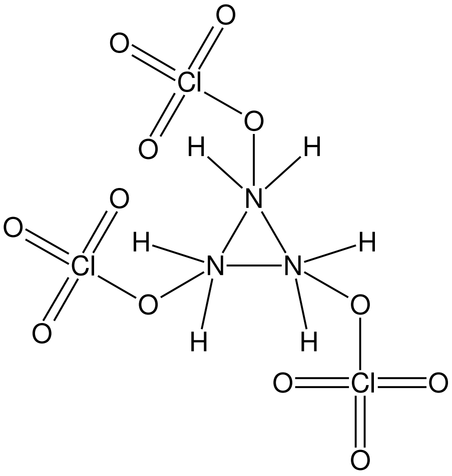
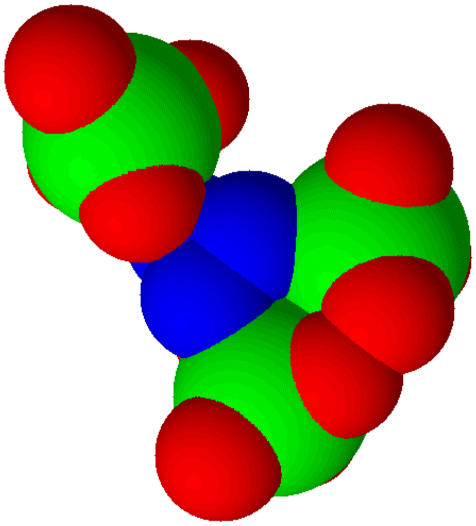
1. 145 grams of triaziridine (prepared in theoretical preparation 39 step 2)	3. 397 grams of freshly prepared lead-II-hydroxide
2. 750 milliliters of dry hexane	

Reaction Equation:



Preparation: Into a standard 3-neck reflux apparatus equipped with motorized stirrer, reflux condenser, thermometer, and seated inside a round bottom heating mantle, pour into this 3-neck reflux apparatus 145 grams of triaziridine (prepared in theoretical preparation 39 step 2), followed by 750 milliliters of dry hexane. Thereafter, pour into the 3-neck reflux apparatus 397 grams of freshly prepared lead-II-hydroxide (prepared by mixing solutions of lead-II-acetate, with sodium or potassium hydroxide), and then begin to rapidly stir the reaction mixture. Immediately thereafter, turn on the heating mantle, and then heat and reflux the reaction mixture under vigorous stirring at 85 Celsius for 8 hours. After 8 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during the cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter-off the insoluble desired product using suction filtration, and then wash with three 750-milliliter portions of warm water, and then suction dry as best as possible. Finally, place the suction dried product mass and place it onto a shallow pan or tray and then allow it to air dry for 48 hours. After 48 hours, scrap off the dried product mass and then store it in amber glass bottles in a cool dry place until use.

Theoretical Preparation 43: triaziridine triperchlorate; Aziridine perchlorate salt; AZP; ADPS; Cl-99

 triazirdine triperchlorate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_6Cl_3N_3O_{12}$
Formula Weight = 346.41954
Composition = H(1.75%) Cl(30.70%) N(12.13%) O(55.42%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = Not available
Nominal Mass = Not available
Average Mass = Not available

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

of refraction):				
7908	Non-flammable. Forms explosive mixtures with powdered metals, and reducing agents.	Very stable (amazing as it seems). Non-sensitive to shock, percussion, pressure, heat, and friction. A secondary explosive; requires blasting cap or detonator for initiation.	Generally non-reactive; shows remarkable stability and non-reactivity; however it very slowly hydrolyzes in water, and explodes in contact with fluorine or when heated with potassium permanganate.	White to off-white crystalline monoclinic crystals or white powder or granules. Melting point: 189 Celsius; Boiling point: begins to decomposes when heated to above 300 Celsius. Soluble in water (1 gram dissolves in 3.6 ml of water at room temperature). Insoluble in all known solvents, but has moderate solubility in liquid ammonia, and remarkably in liquid nitrogen

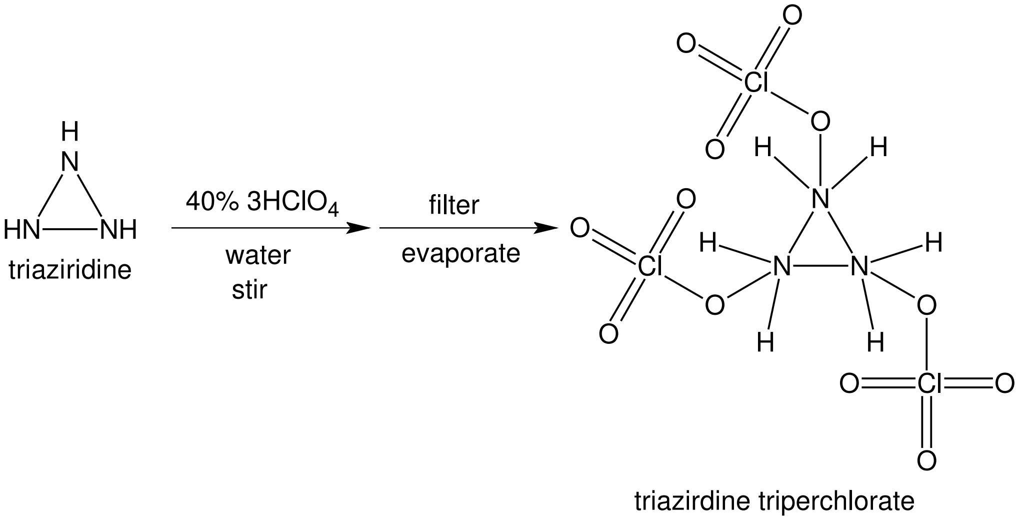
Estimated Compound summary: triaziridine triperchlorate is a very simple compound to make and has remarkable properties and stability. It has excellent physical properties making it useful in melting and casting into explosive munitions of all kinds including mortar shells, artillery shells, missile warheads, and shape charges; however, its properties make it one of the best oxidizers in high performance solid rockets fuels. triaziridine triperchlorate has a greater weight to thrust ratio then any other oxidizer in use. triaziridine triperchlorate when admixed with powered aluminum and cured with epoxy forms a high performance rocket fuel with a thrust to weight ration three times greater then the more common ammonium perchlorate. triaziridine triperchlorate has use in making high explosive munitions, but when molten it is insoluble in all known molten explosives including TNT, RDX, HMX, ect, ect,. triaziridine triperchlorate is designed to be more of an oxidizer in high performance rocket fuels, high performance gun propellants, and pyrotechnic mixtures rather then used as a explosive; however it does have normal physical attributes for making explosive munitions—the decision of its use is based on the manufacturer. triaziridine triperchlorate makes outstanding pyrotechnic mixtures for making fireworks, flares, incendiary agents, and smoke grenades. triaziridine triperchlorate is one of the best oxidizers for use in making a variety of high performance rocket fuels, high performance gun propellants, and pyrotechnics. In another note, triaziridine triperchlorate can be melted down and poured into molds for making powerful shape charges do to its high gas ratio and can be used in a variety of commercial and military uses. triaziridine triperchlorate can also be melted down or dead pressed under high pressure for making demolition blocks for a variety of commercial and military operations. Note: triaziridine triperchlorate should no be used in underground mining operations due to toxic fumes produced upon detonation.

Safety Info: Use extreme caution when handling perchloric acid of any concentration as it has a tendency to catch organic material on fire. Wear gloves and avoid skin contact of perchloric acid, which can produce painful blisters on the skin.

Materials:

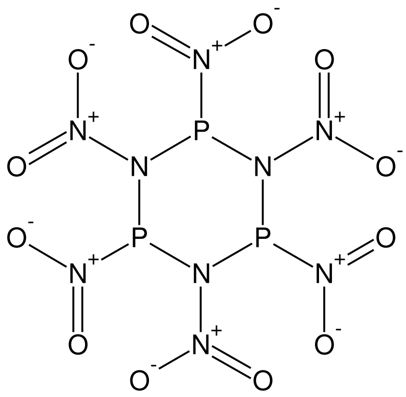
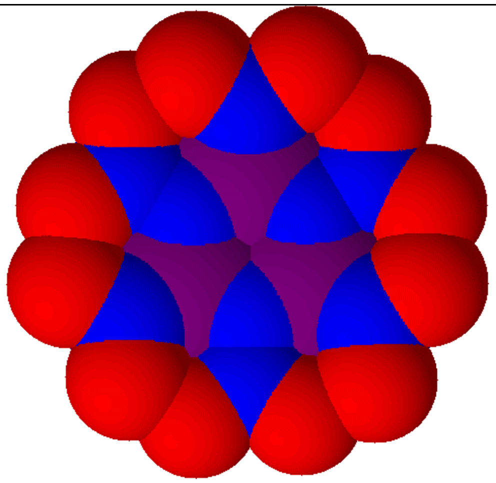
1. 209 grams of triaziridine (prepared in theoretical preparation 39, step 2)	2. 1399 grams of 98% perchloric acid or 2000 grams of 70% perchloric acid

Reaction Equation:



Preparation: Into a large 6-liter beaker, place this 6-liter beaker inside a 8-liter container filled with ice, and then station a electric motorized stirrer settled in the center of the 6-liter beaker and held by a holding clamp attached to a base support rod, pour into the 6-liter beaker 209 grams of triaziridine (prepared in theoretical preparation 39 step 2). Immediately thereafter, turn on the motorized stirrer and add in 1500 milliliters of ice cold water, and then begin to rapidly stir the triaziridine/water mixture. Thereafter, slowly pour in 3500 grams of a 40% perchloric acid solution (prepared by slowly adding and dissolving 1399 grams of 98% perchloric acid into 2102 milliliters of ice cold water. Note: commercial perchloric acid is mostly sold as a 70% solution, so dilute 2000 grams of 70% perchloric acid with 602 milliliters of cold water) into the 6-liter beaker containing the triaziridine/water reaction mixture under rapid agitation, while maintaining the reaction mixture at a temperature below 30 Celsius. After the addition of the perchloric solution, continue to rapidly stir the reaction mixture and maintain its temperature below 30 Celsius for 4 hours. Note: the entire addition of the perchloric acid should take about 2 hours. Note: more ice may have to be added to the outer 8-liter container as the previous ice may have melted during the addition of the perchloric acid. After 4 hours, remove the 8-liter ice bath container, and allow the reaction mixture in the 6 liter beaker to warm to room temperature if not already done so. Thereafter, filter the entire reaction mixture using suction filtration to remove the insoluble impurities, and then place the filtered reaction mixture into a large rotary evaporator, and evaporate-of the water under high vacuum and at a temperature around 75 to 80 Celsius. Continue to evaporate-off the water until dry solid remains. When dry solid remains, turn off the rotary evaporator, remove the vacuum, and then allow the left over residue solid in the rotary evaporator to cool to room temperature. Once it has recover the desired product of triaziridine triperchlorate and then store in a airtight container in a cool dry place until use.

Theoretical Preparation 44: 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane; Nitro triazatriphosphinane; HTATP; Cl-109

 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane	
Expanded structure	3D structure

Molecular data:
Molecular Formula = $N_9O_{12}P_3$
Formula Weight = 410.974386
Composition = N(30.67%) O(46.72%) P(22.61%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 410.887924 Da
Nominal Mass = 411 Da
Average Mass = 410.9744 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
10,009; estimated by infrared spectroscopy	Non-flammable under nominal conditions; will burn without significant violence when heated to 250 Celsius and ignited using a flame. Reacts and burns violently when admixed with powdered metals or reducing agents.	Very stable; very stable towards heat, friction, shock, percussion, and pressure. A secondary explosive. Requires blasting cap or detonator for initiation.	Virtually inert to chemical attack. Does not react with strong oxidizers or reducing agents even when heated; however, can be reduced by heating with concentrated hydrochloric acid in the presence of finely divided Tin. Explodes in contact with	White off-white crystalline masses, chunks, granules, or powder. Impure forms will have a yellowish tint. Melting point 290 Celsius; boiling point: decomposes when heated to above 350 Celsius, and decomposes

			fluorine.	vigorously when heated to 410 Celsius. Practically insoluble in water and most organic solvents. Soluble in concentrated sulfuric acid, liquid ammonia, and liquid sulfur dioxide.

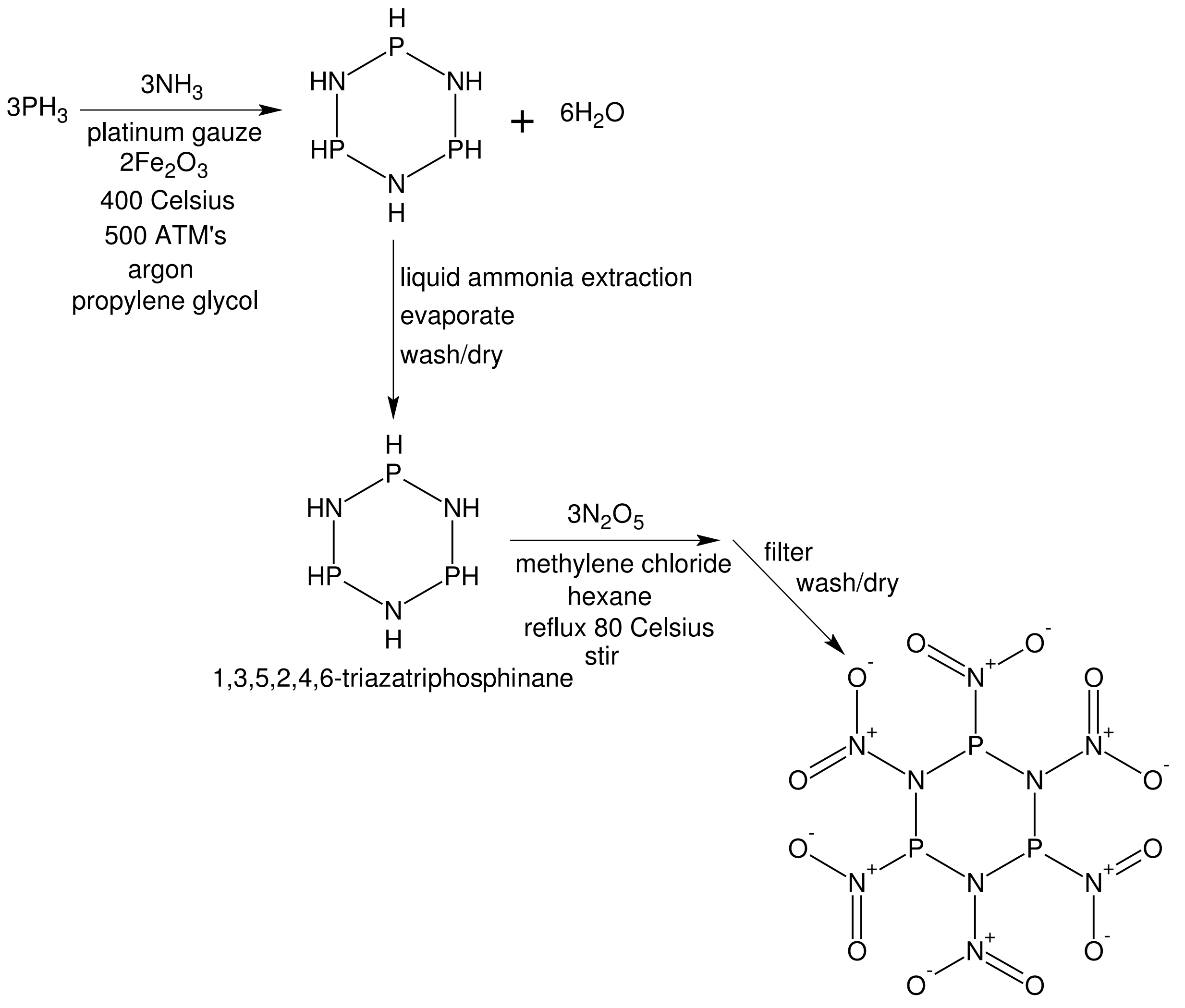
Estimated Compound summary: 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane is a very powerful explosive that has numerous uses in manufacturing explosive munitions. It is very stable and with its high melting point, it makes for a perfect high explosive with numerous uses. 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane can be melted down and casted into shape charges for making powerful anti-tank weapons, or for use in commercial and military demolition operations. Molten 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane is insoluble in most other explosives, so it is used mostly by itself when melting and casting is desired. 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane can be dead pressed into explosive bodies under a pressure of 3000 psi, or it can be melted down and then poured into explosive munitions of all imaginable types ranging from mortar shells, artillery shells, high velocity projectiles, missile warheads, and practically any explosive munition(s). 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane reacts violently when mixed with reducing agents and ignited with a spark or flame, hence it can be used to manufacturing powerful high performance gun propellants, and high performance rocket propellants (the latter mostly when mixed with powered aluminum and cured with epoxy). Warning! When using 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane in making high performance gun propellants caution should exercised as these propellants are very powerful and can lead to weapon malfunction when the weapon is fired (the weapons firing the ammo utilizing 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane may explode), so multiple experiments and tests should be used to determine burn (deflagration) rates and weight of propellants used in the cartridge. Due to the danger of 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane compositions, pyrotechnic compositions containing the said compound should be carried out with great care and effort.

Safety Info: Use care, and wear proper gloves and lab clothing when handling mercury, which is very toxic and can lead to death if ingested, inhaled, or absorbed through the skin. Use extreme care and wear gloves and eye protection when handling metallic sodium, which is very toxic, and explodes in contact with water. Use great care when handling phosphine gas, which is spontaneously combustible in the presence of oxygen. Use proper ventilation and wear gloves and eye protection when handling ammonia gas, which is highly irritating to the nose, throat, and eyes—use the same caution when handling liquid ammonia. Extinguish all sources of ignition when handling hexane, which is highly volatile and flammable. Wear gloves, eye protection, and proper lab clothing when dealing with dinitrogen pentoxide, and keep it out of contact with combustible materials.

Materials:

1. 500 milliliters of propylene glycol	8. 51 grams of dry ammonia gas
2. 718 grams of freshly prepared iron-III-oxide	9. 2250 milliliters of liquid ammonia
3. 5 grams of platinum gauze	10. 600 milliliters of hexane
4. 750 milliliters of pure liquid mercury	11. 326 grams of dinitrogen pentoxide
5. 150 grams of metallic sodium	12. 1400 milliliters of methylene chloride
6. Argon gas from tank	13. 1500 milliliters of 95% ethyl alcohol
7. 101 grams of dry phosphine gas	

Reaction Equation:

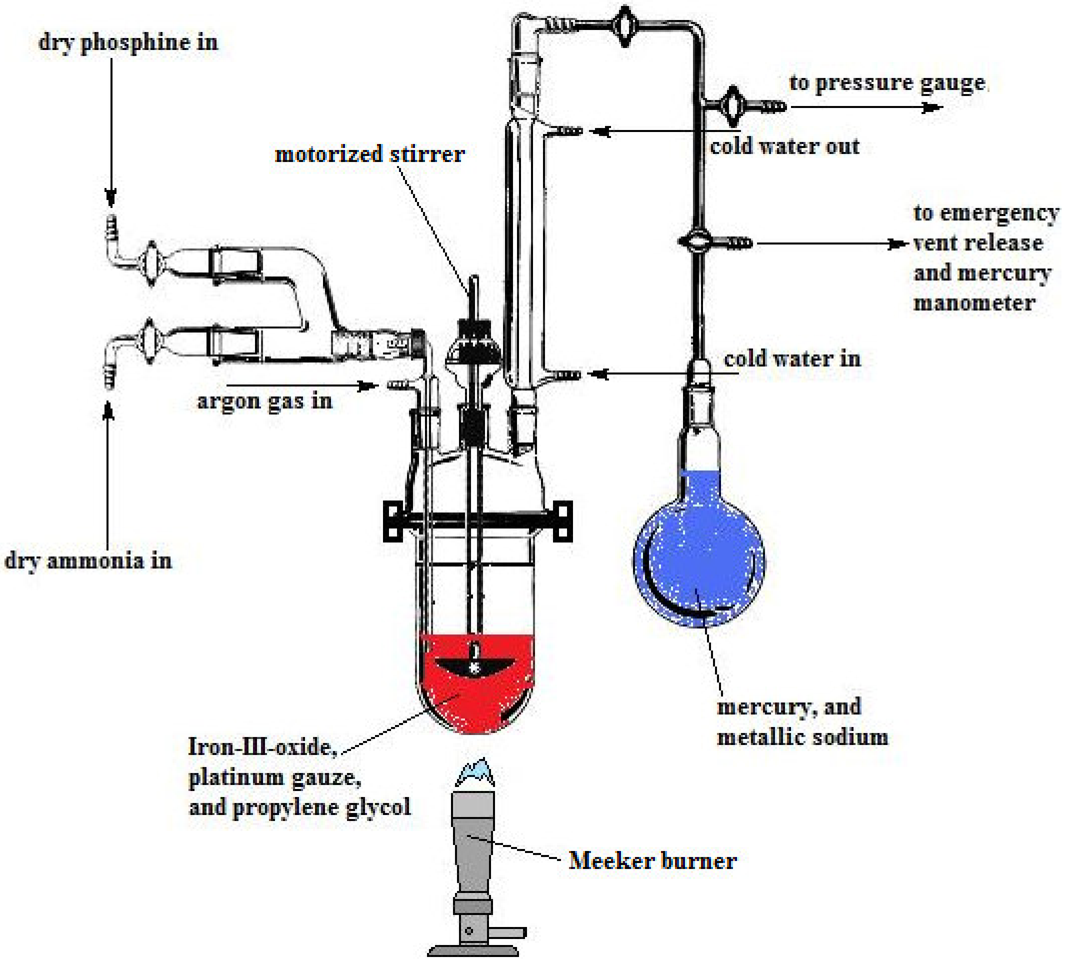


1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane

Preparation:

Step 1: Preparation of 1,3,5,2,4,6-triazatriphosphinane

Into an apparatus as illustrated, place 500 milliliters of propylene glycol into the reaction flask, followed by 718 grams of freshly prepared iron-III-oxide, and then followed by 5 grams of platinum gauze. Now, as illustrated below, into the appropriate flask, add in 750 milliliters of pure liquid mercury, and then add in 150 grams of metallic sodium to this mercury. Thereafter, begin an argon purge and bring the pressure of the apparatus system to 500 ATM's. When the pressure inside the apparatus system has reached 500 ATM's, ignite the meeker burner and begin to heat the contents in the reaction flask to 400 Celsius with constant stirring. When the pressure inside the apparatus system is at a constant 500 ATM's, and its temperature reaches 400 Celsius, begin to bubble into the reaction mixture dry phosphine at a flow rate of 95 mg per/minute, and at the same time begin to bubble into the reaction mixture dry ammonia gas at a flow rate of 45 mg per/minute while stirring the reaction mixture and maintaining a heat and pressure of 400 Celsius at 500 ATM's. Continue to bubble into the reaction mixture dry phosphine gas and dry ammonia gas under a pressure of 500 ATM's and at a temperature of 400 Celsius under rapid stirring until 101 grams of dry phosphine gas and 51 grams of dry ammonia gas has been added. Once 51 grams of dry ammonia gas, and 101 grams of dry phosphine gas has been added, turn off both gas sources (usually from tank), close both the stopcocks near the entry points, and then continue to rapidly blend the reaction mixture under a pressure of 500 ATM's and at a temperature of 400 Celsius under rapid stirring for 6 hours. After 6 hours, turn off the meeker burner, release the pressure gauge and allow the reaction mixture to come down to standard atmospheric pressure and a temperature of room temperature. Thereafter, continue to stir the reaction under standard atmospheric temperature and at a temperature of room temperature for 4 hours. After 4 hours, stop the stirring, disassemble the apparatus, and then filter-off, using suction filtration, the insoluble desired product, which will also contain elemental iron and platinum gauze. Note: use tweezers to pluck-out the platinum gauze and save for another procedure. The iron can be recycled or disposed of. Now, take the filter cake remaining after suction filtration (which will also contain finely divided iron which will begin to oxidize, and place this filter cake into a large beaker and then extract the filter cake with three 750-milliliter portions of liquid ammonia (see extraction for proper method). After each extraction combine all three extracts if not already done so, and then pour this combined extract mixture onto a shallow pan, and allow it to evaporate at room temperature. Once all the liquid ammonia has evaporated, and dry solid remains, scrape off and collect the dried residue and then place this dry residue into a clean Buchner funnel fitted with standard filter paper, turn on the vacuum (suction filtration), and then wash this collected residue with three 750-milliliter portions of hot water, and then suction dry as best as possible. Finely, take this washed desired product, and place onto a clean shallow pan, and allow it to thoroughly air-dry for 3 days. After 3 days, recover the dried mass, and save for step 2.


dry phosphine in
to pressure gauge
motorized stirrer
cold water out
to emergency vent release and mercury manometer
cold water in
argon gas in
dry ammonia in
mercury, and metallic sodium
Iron-III-oxide, platinum gauze, and propylene glycol
Meeker burner

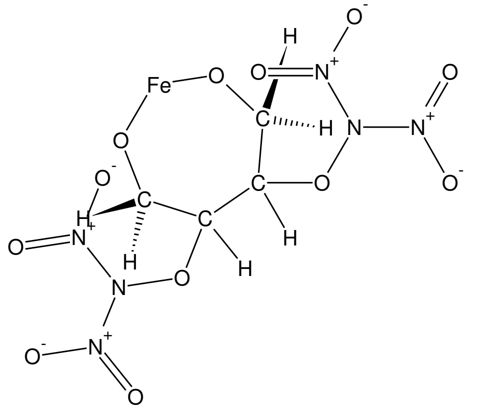
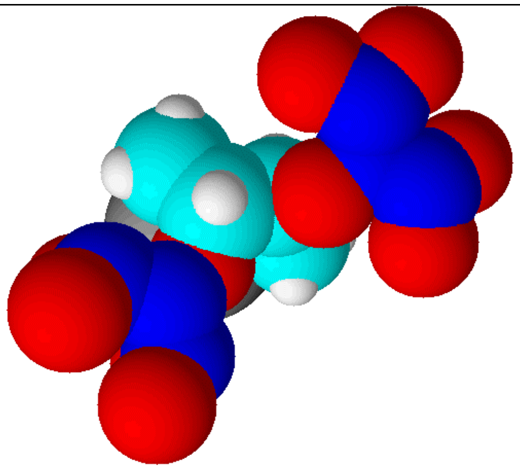
All Nickel setup for preparing 1,3,5,2,4,6-triazatriphosphinane

Step 2: Preparation of 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane

Into a large 3-neck flask sitting inside a round bottom heating mantle, and equipped with cold-water condenser, addition funnel, thermometer, and motorized stirrer, pour in 600 milliliters of hexane, and then add in the 1,3,5,2,4,6-triazatriphosphinane obtained in step 1. Then begin to stir this mixture, and then turn on the heating mantle and bring the temperature of the contents in the 3-neck flask to around 80 Celsius under reflux. Immediately thereafter pour into the addition funnel 1/3rd of a mixture obtained by dissolving 326 grams of dinitrogen pentoxide into 1400 milliliters of methylene chloride. Once the contents in the 3-neck flask begins to boil under reflux, begin a moderate drip of the 1/3rd portion of dinitrogen pentoxide/methylene chloride mixture from the addition funnel while vigorously stirring the reaction mixture and maintaining its temperature around 80 Celsius. Once all this 1/3rd portion of dinitrogen pentoxide/methylene chloride mixture has been added to the reaction mixture from the addition funnel, pour in another 1/3rd of the dinitrogen pentoxide methylene chloride mixture into the addition funnel, and then continue a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while vigorously stirring the reaction mixture and maintaining its temperature under reflux at 80

Celsius. Once all this dinitrogen pentoxide/methylene chloride has been added to the reaction mixture, pour in the final 1/3rd of the dinitrogen pentoxide/methylene chloride mixture into the addition funnel, and then continue a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while vigorously stirring the reaction mixture and maintaining its temperature under reflux at 80 Celsius. Once all the dinitrogen pentoxide/methylene chloride has been added, continue to vigorously stir the reaction mixture and maintain a reflux at 80 Celsius for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during this cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter-off the insoluble desired product of 1,2,3,4,5,6-hexanitro-1,3,5,2,4,6-triazatriphosphinane using suction filtration, and then wash this filtered-off desired product with three 500-milliter portions of 95% ethyl alcohol, and then wash with three 750-milliliter portions of hot water, and then suction dry as best as possible. Finally, take this suction dried desired product and place it onto a shallow pan or tray and allow it to air dry for 48 hours. After 48 hours the desired product should be scraped off the pan or tray and placed into an airtight container and stored in a cool dry place until use.

Theoretical Preparation 45: ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine; DNAODFDN; K12-Iron salt; V-90

 ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_4H_6FeN_6O_{12}$
Formula Weight = 385.96844
Composition = C(12.45%) H(1.57%) Fe(14.47%) N(21.77%) O(49.74%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = 5.657 ± 0.299 g/cm^3
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = Not available
Nominal Mass = Not available
Average Mass = Not available

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

of refraction):				
8,780	Non-flammable; detonates when touched by a spark or flame.	Stable at room temperature and temperatures up to 190 Celsius. Relatively stable to pressure, but moderately stable to shock, percussion, and friction. A sample will detonate when struck with a hammer or hit by a rifle bullet. A sample will also detonate when heated to above 191 Celsius, touched by heated Nichrome wire, or electric arc. A primary explosive.	Relatively non-reactive; very slowly hydrolyzes by water, and is slowly oxidized when exposed to the atmosphere. Reacts violently often with detonation when treated with halogens, ozone, strong oxidizers, or powdered metals.	Dark grayish black crystalline powder with a metallic sheen. Melting point: N/A; Boiling point: N/A. Insoluble in water and most organic solvents. Barely soluble in liquid ammonia, but has slight solubility in carbon disulfide.

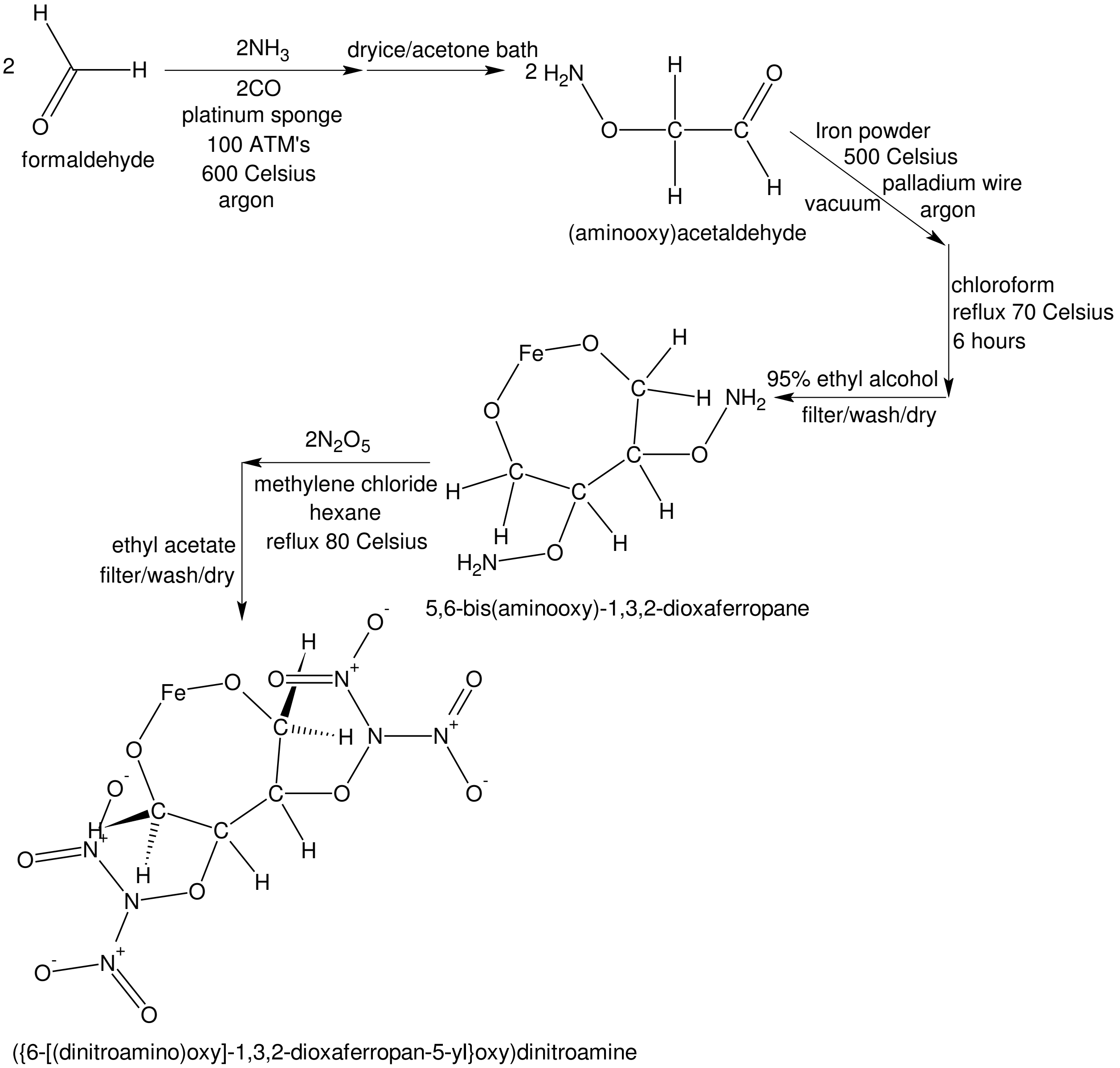
Estimated Compound summary: ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine is a new and radical and interesting primary explosive. It has interesting characteristics and properties making it well suitable for use as a replacement for lead azide, mercury fulminate, diazodinitrophenol, and other primary explosives for use in blasting caps and detonators. It also has much greater stability then the aforementioned primary explosives, and can be stored in use for many years without any worry of deteriation or premature detonation. ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine has the highest detonation velocity of any known primary explosives, hence smaller quantities of this explosive are needed in blasting caps and detonators making a valuable primary explosive for priming secondary explosives for multiple uses including priming shape charges, and detonating cord. ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine is an excellent primary explosive that can be used in fuses to prime a variety of explosive munitions including mortar shells, artillery shells, missile warheads, rockets, shape charges of various sizes including linear shape charges, and because of its general water resistance (only very slowly hydrolyzes in water) it can be used with stability in munitions that are exposed to the elements such as land mines and torpedoes. Also, do to its general stability ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine does not need to be diluted with an inert filler for stability it can be carefully and actually dead pressed under 1000 psi into blasting caps and detonators for fuses or for general blasting caps for priming of demolition explosives for commercial and military operations of all kinds. ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine can also be used to replace PETN in manufacturing detonating cord that does not have to be primed like PETN detonating cords, and can be initiated by a simple flame or by electrical means forming a powerful detonating cord, even more powerful then the more common PETN detonating cord. ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine is rather un-explored and shows remarkable properties for use in a variety of explosive munitions for a wide variety of explosive operations, so this compound does warrant further investigation. It use in pyrotechnic compositions is unknown, but may demonstrate further investigation.

Safety Info: Use maximum ventilation and wear proper gloves and eye protection when handling formaldehyde gas, carbon monoxide, and ammonia gas. Ammonia gas is highly irritating to the eyes, nose, and throat. Formaldehyde gas is a narcotic in high concentrations and is toxic. Carbon monoxide is a deadly poison so use extreme care. Use proper ventilation when handling chloroform, which is a narcotic in moderate concentrations, avoid inhalation of the vapors. Extinguish all flames and sources of ignition when handling hexane, which is highly volatile and flammable. Wear proper gloves, eye protection, and proper lab clothing when handling dinitrogen pentoxide, and avoid contact with combustible materials. Use ventilation when handling ethyl acetate, which is a narcotic in high concentrations.

Materials:

1. 50 grams of platinum sponge	8. 500 milliliters of chloroform
2. 57 grams of formaldehyde gas	9. 1250 milliliters of 95% ethyl alcohol
3. 53 grams of carbon monoxide gas	10. 500 milliliters of hexane
4. 32 grams of ammonia gas	11. 204 grams of dinitrogen pentoxide
5. Argon gas from tank	12. 1000 milliliters of methylene chloride
6. 53 grams of powdered iron	13. 500 milliliters of ethyl acetate
7. 5 grams of palladium wire of 20 gauge	

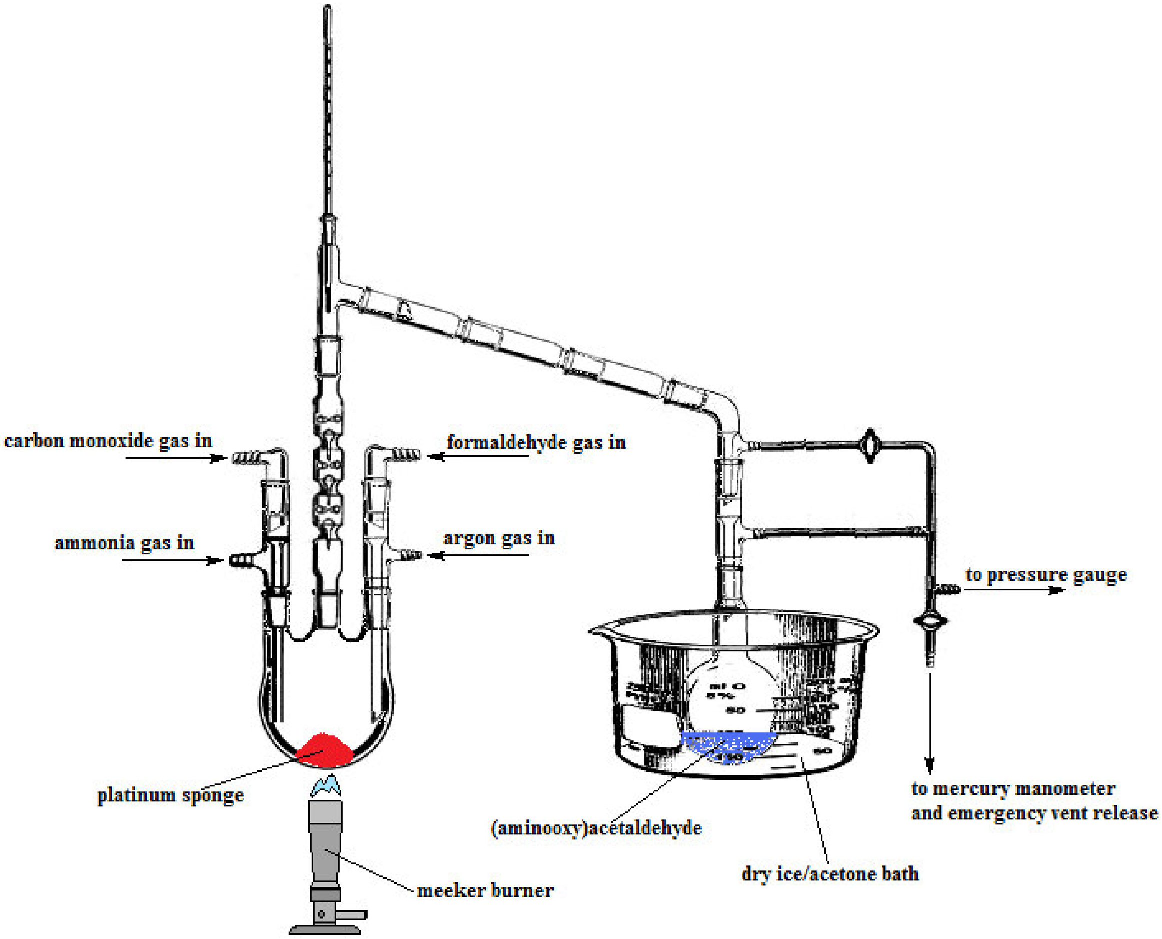
Reaction Equation:



Preparation:

Step 1: preparation of (aminooxy)acetaldehyde

Assemble an apparatus as similar to the one listed below, and then place 50 grams of platinum sponge into the reaction flask as illustrated below. Note: 50 grams of platinum sponge is an enormous amount of platinum with a heavy cost, but it is inert and can be reused thousands of times; therefore, the initial investment will pay off. Thereafter, seal the apparatus, and then begin a argon gas purge under a flow rate of 150 mg per/minute and bring the pressure inside the apparatus to around 100 ATMs. When the pressure inside the apparatus reaches around 100 ATM's, turn on the meeker burner and heat the platinum sponge and reaction flask to 600 Celsius. Now, when the temperature and pressure inside the reaction flask reaches 100 ATM's and 600 Celsius, begin to pass into the reaction flask formaldehyde gas at a flow rate of 45 mg per/minute, simultaneously begin to pass into the reaction flask carbon monoxide gas at a flow rate of 42 mg per/minute, and simultaneously begin to pass into the reaction flask ammonia gas at a flow rate of 80 mg per/minute while heating the reaction flask at 600 Celsius under a pressure of 100 ATM's. Continue to pass into the reaction flask formaldehyde gas, carbon monoxide gas, and ammonia gas at their respective flow rates until 57 grams of formaldehyde gas has been passed in, and 53 grams of carbon monoxide gas has been passed in, and 32 grams of ammonia gas has been passed in, while maintaining a argon purge and a reaction temperature of around 600 Celsius under 100 ATM's. Once all the formaldehyde gas, carbon monoxide gas, and ammonia gas has been passed into the reaction flask, turn-off the valves to the gasses sources, but continue to purge the apparatus with argon gas at a pressure of 100 ATM's but turn off the meeker burner and allow the platinum sponge to cool to room temperature while maintaining an argon purge. Note: during the reaction process, the solid desired product of (aminooxy)acetaldehyde will form, and will volatize and be carried over to the receiver flask submerged in a dry ice/acetone bath, where-by it will solidify to a messy mass. Now, once the platinum sponge has cooled, disassemble the apparatus if desired, and then remove the receiver flask containing the desired product of (aminooxy)acetaldehyde from the dry ice/acetone bath, and allow it to warm to room temperature, and then save for step 2.



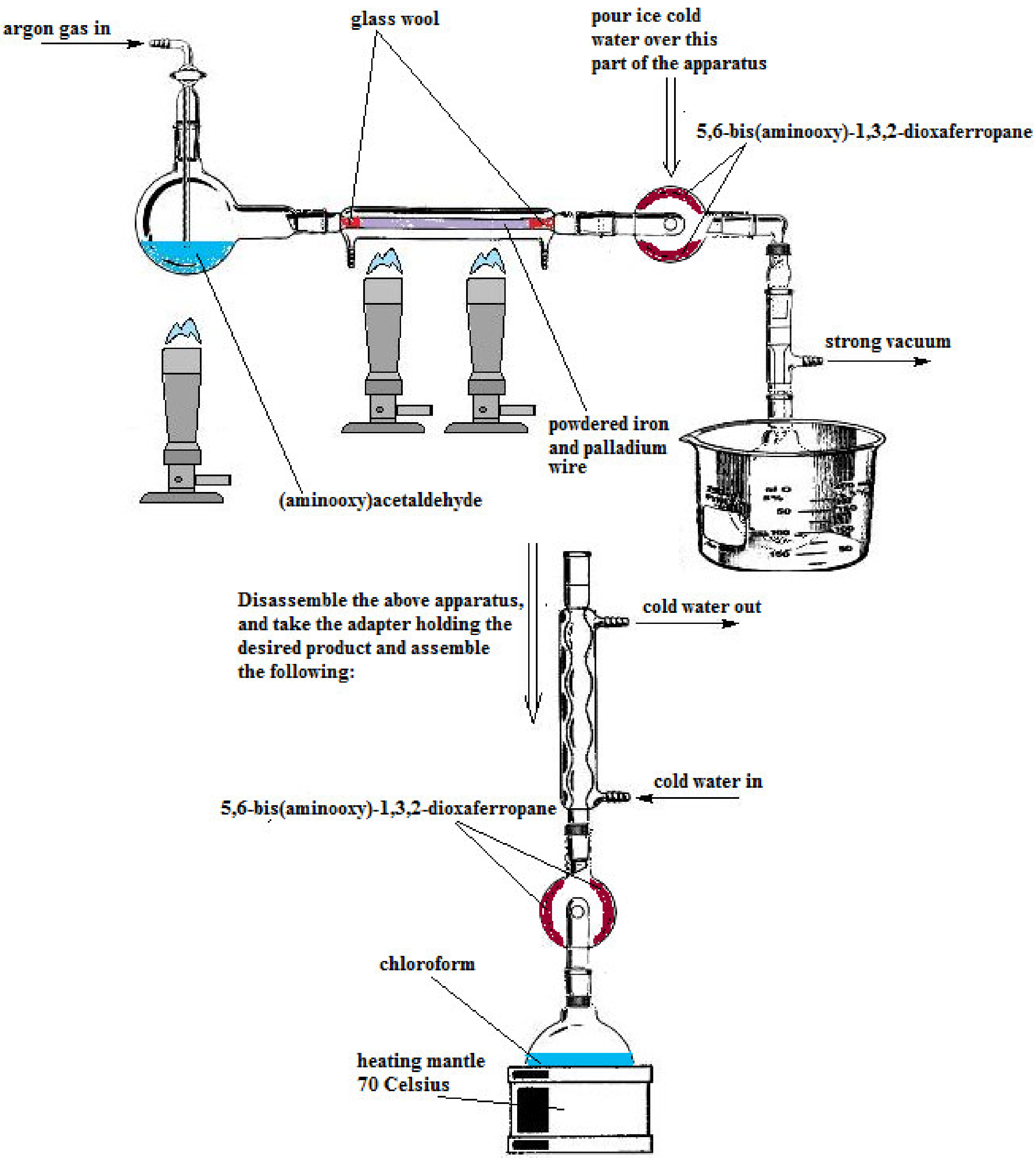
All Nickel apparatus for preparing (aminooxy)acetaldehyde.

Step 2: preparation of 5,6-bis(aminooxy)-1,3,2-dioxaferropane

Into an apparatus as illustrated below, place the messy mass product obtained in step 1 into the flask as illustrated. Then place 53 grams of powdered iron and 5 grams of palladium wire of 20 gauge into the condenser, and then seal both ends with glass wool as illustrated. Note: shake the condenser to allow the iron powder to lay out flat and evenly across the surface of the inner tube. Thereafter, begin a argon purge by passing argon gas at a flow rate 150 mg per/minute, and turn on the vacuum pump (not illustrated) and produce a strong vacuum. Once a strong vacuum is achieved, turn on all three meeker burners and heat the parts of the apparatus as illustrated at a temperature of 500 Celsius under high vacuum. Immediately thereafter, begin to pour ice-cold water over the round adapter as illustrated below, and then continue to pour fresh ice cold water over this round adapter throughout the reaction. Continue to purge the apparatus with argon gas, and continue to heat the parts of the apparatus as illustrated below at 500 Celsius until all the messy mass of (aminooxy)acetaldehyde (prepared in step 1) has disappeared from the flask containing it as illustrated below. Once all the (aminooxy)acetaldehyde no longer occupies the right flask as illustrated below, turn off the vacuum and turn off all three meeker burners, but continue to purge the apparatus with argon until the entire apparatus has cooled to room temperature. Note: during this cool down period continue to pour ice-cold water over the round adapter as illustrated below. Once the apparatus has cooled to room temperature, turn off the argon purge, and stop pouring ice cold water over the round adapter as illustrated below. Note: the desired product of 5,6-bis(aminooxy)-1,3,2-

dioxaferropane will be stuck to the walls of the round adapter as illustrated below. Now, disassemble the apparatus and then take the round adapter containing the 5,6-bis(aminooxy)-1,3,2-dioxaferropane stuck to its inside wall and assemble the apparatus as illustrated in the second illustration below.

Now, into the round bottom single neck flask pour in 500 milliliters of chloroform, and then assemble the apparatus as illustrated in the second illustration below. Thereafter, begin to pump in cold water into the bulb condenser, and then turn on the heating mantle to 70 Celsius, and begin a reflux. Now, continue to reflux the chloroform for 6 hours at 70 Celsius. Note: during the reflux process, the desired product of 5,6-bis(aminooxy)-1,3,2-dioxaferropane (stuck to the inner walls of the round adapter) will slowly dislodge and partially dissolve in the chloroform and will travel down to the round bottom single neck flask contained in the heating mantle as illustrated in the second illustration below. After 6 hours, all of the desired product of 5,6-bis(aminooxy)-1,3,2-dioxaferropane should have been dislodge or partially dissolved in the chloroform, so after 6 hours, turn off the heating mantle and allow the chloroform/5,6-bis(aminooxy)-1,3,2-dioxaferropane to cool to room temperature. Once it has, disassemble the apparatus, and then pour the chloroform/5,6-bis(aminooxy)-1,3,2-dioxaferropane into a large suitable sized beaker, and then pour in 500 milliliters of 95% ethyl alcohol. At this point any and all of the desired product of 5,6-bis(aminooxy)-1,3,2-dioxaferropane will separate out, and settle to the bottom. Note: after adding in the alcohol, stir the chloroform/ethyl alcohol/5,6-bis(aminooxy)-1,3,2-dioxaferropane mixture for 2 hours at room temperature. After 2 hours, filter-off the desired product of 5,6-bis(aminooxy)-1,3,2-dioxaferropane using suction filtration, and then wash with three 250-milliliter portions of fresh 95% ethyl alcohol, and then wash with three 500-milliliter portions of warm water, and then suction dry as best as possible. Finely, place the suction dried washed product of 5,6-bis(aminooxy)-1,3,2-dioxaferropane and place it into a dessicator over concentrated sulfuric acid for 2 weeks. After 2 weeks, the desired dried product of 5,6-bis(aminooxy)-1,3,2-dioxaferropane is ready for step 3.

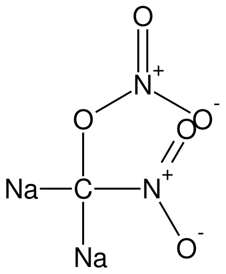
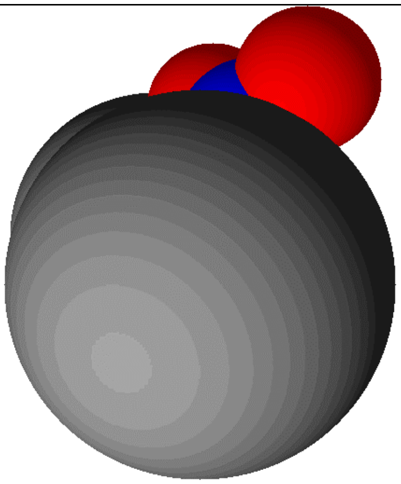

argon gas in
glass wool
pour ice cold water over this part of the apparatus
5,6-bis(aminooxy)-1,3,2-dioxaferropane
strong vacuum
powdered iron and palladium wire
(aminooxy)acetaldehyde
Disassemble the above apparatus, and take the adapter holding the desired product and assemble the following:
cold water out
cold water in
5,6-bis(aminooxy)-1,3,2-dioxaferropane
chloroform
heating mantle 70 Celsius

The first apparatus is made up of all nickel except for the lower portion. The lower apparatus is made of glass except for round adapter containing the desired product stuck to the inside part of the walls, this nickel adapter is tapered to fit 24/40 glass, which is what the lower apparatus is made up of. Note: as the chloroform boils, it will dislodge and partially dissolve the desired product of 5,6-bis(aminooxy)-1,3,2-dioxaferropane from the round upper adapter wall.

Step 3: preparation of ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine

Into a moderate sized 3-neck flask sitting inside a round bottom heating mantle, and equipped with cold-water condenser, addition funnel, thermometer, and motorized stirrer, pour in 500 milliliters of hexane, and then add in the 5,6-bis(aminooxy)-1,3,2-dioxaferropane obtained in step 2. Then begin to stir this mixture, and then turn on the heating mantle and bring the temperature of the contents in the 3-neck flask to around 80 Celsius under reflux. Immediately thereafter pour into the addition funnel ½ of a mixture obtained by dissolving 204 grams of dinitrogen pentoxide into 1000 milliliters of methylene chloride. Once the contents in the 3-neck flask begins to boil under reflux, begin a moderate drip of the ½ portion of dinitrogen pentoxide/methylene chloride mixture from the addition funnel while vigorously stirring the reaction mixture and maintaining its temperature around 80 Celsius. Once all this ½ portion of dinitrogen pentoxide/methylene chloride mixture has been added to the reaction mixture from the addition funnel, pour in the last ½ of the dinitrogen pentoxide methylene chloride mixture from the addition funnel, and then continue a moderate drip of this dinitrogen pentoxide/methylene chloride mixture into the reaction mixture while vigorously stirring the reaction mixture and maintaining its temperature under reflux at 80 Celsius. Once all this dinitrogen pentoxide/methylene chloride has been added to the reaction mixture, continue to vigorously stir the reaction mixture and maintain a reflux at 80 Celsius for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during this cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a large suitable sized beaker, and then pour in 500 milliliters of ethyl acetate, and then stir this new mixture for 4 hours at room temperature. After stirring this new mixture for 4 hours at room temperature, filter-off the insoluble product using suction filtration, and then wash this filtered-off desired product with three 750 milliliter portions of warm water, and then suction dry as best as possible. Finally place the filtered-off suction dried product of ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine into a dessicator over concentrated sulfuric acid for 2 weeks. After 2 weeks, the dried desired product of ({6-[(dinitroamino)oxy]-1,3,2-dioxaferropan-5-yl}oxy)dinitroamine is ready for use. Note: this desired product should be stored in the dessicator until use.

Theoretical Preparation 46: µ-[nitro(nitrooxy)methylidene]disodium; Nitroxy sodium; Nitro nitrooxy sodium; Sodium-NNM; PL-107

 µ-[nitro(nitrooxy)methylidene]disodium	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $CN_2Na_2O_5$

Formula Weight = 166.000638

Composition = C(7.24%) N(16.87%) Na(27.70%) O(48.19%)

Molar Refractivity = Not available

Molar Volume = Not available

Parachor = Not available

Index of Refraction = Not available

Surface Tension = Not available

Density = Not available

Dielectric Constant = Not available

Polarizability = Not available

Monoisotopic Mass = 165.960261 Da

Nominal Mass = 166 Da

Average Mass = 166.0006 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

mass and index of refraction):				
4599, estimated by NMR spectra	Flammable solid; may ignite spontaneously in moist air. Burns forming carbon monoxide and nitrogen oxides—never use in enclosed areas.	Amazingly relatively stable to heat, pressure, and friction, but sensitive to shock and percussion, but more stable then lead azide or mercury fulminate. A primary explosive.	Highly reactive. Reacts violently often with explosions in contact with water. Explodes when treated with halogens or strong oxidizing agents. Forms shock sensitive and heat sensitive compositions that explode when mixed with reducing agents and ignited, or upon shock, or friction.	Silvery-white decaclinic mono-lithic crystalline mass. Melting point: Explodes when heated to above 109 Celsius. Boiling point: N/A; insoluble in most organic solvents, but has moderate solubility in liquid ammonia.

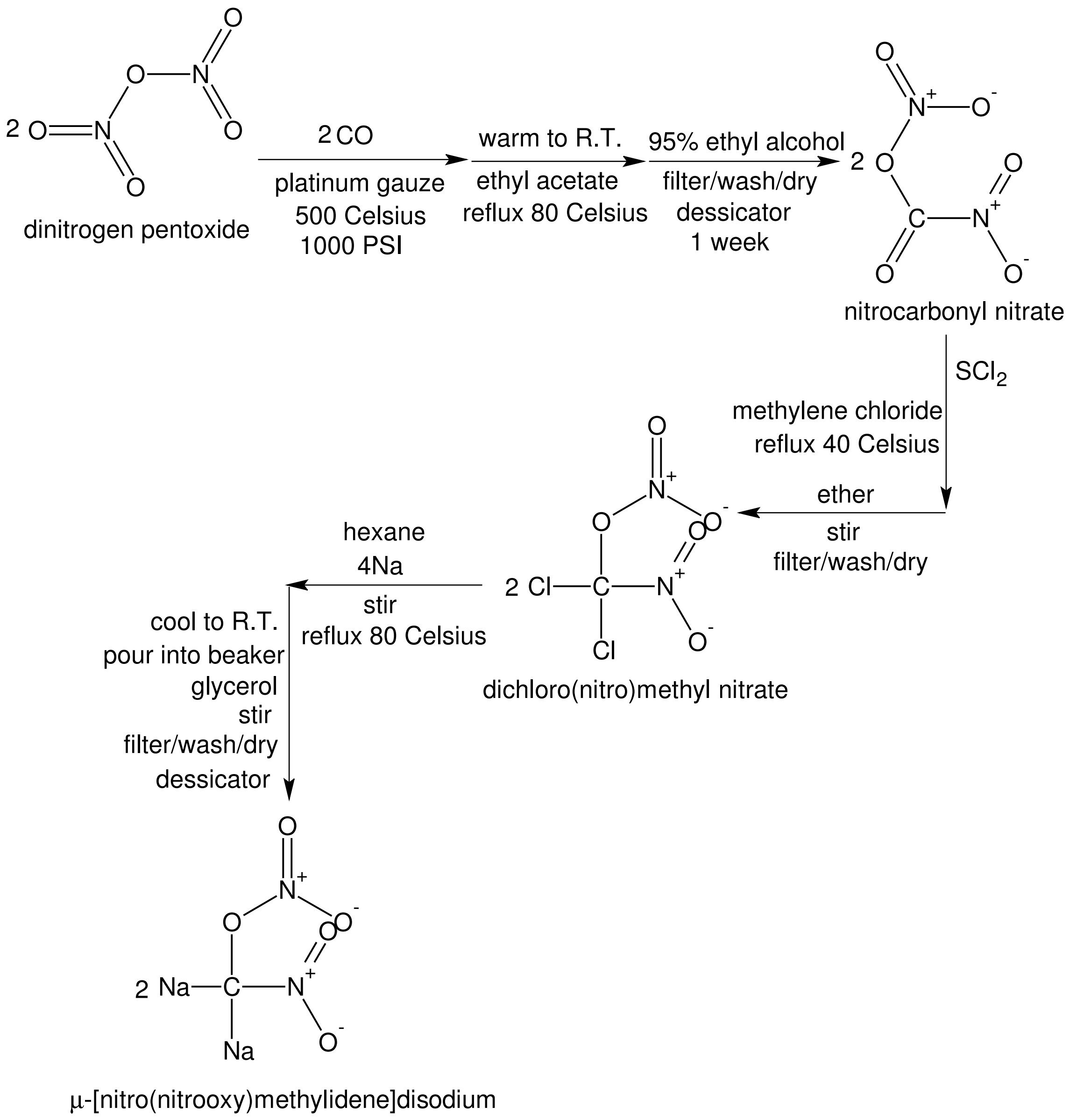
Estimated Compound summary: μ-[nitro(nitrooxy)methylidene]disodium is a remarkable yet dangerous primary explosive that ultimately has limited use in the explosives industry; however, it is listed in this book because when used properly it demonstrates great potential for making stable and a unique new blend of high explosives never seen before. When μ-[nitro(nitrooxy)methylidene]disodium is carefully mixed with a granulated explosive such as ammonium nitrate, TNT, PETN, RDX, HMX, ect., and then the resulting mixture mixed thoroughly with a high energy plasticizer, it forms a uniform putty-like high explosive that can be molded and shaped into any desired shape or form, which can be easily be detonated by a flame or electric arc or heated Nichrome wire. These putty-like explosive compositions can be used in place of C4 for molding and shaping for a variety of uses including making improvised explosive devices, shape charges, and many other forms of explosive devices. Also, these putty-like explosives are resistant to moisture and water, and hence can be used alone by themselves in explosive operations for military use, or dead pressed into explosive shells for mortars and artillery, or for making missile warheads, anti-tank warheads, rocket warheads, and land mines. μ-[nitro(nitrooxy)methylidene]disodium has limited use in manufacturing blasting caps and detonators, unless the blasting caps and detonators are permanently sealed airtight and water tight. Other then that, μ-[nitro(nitrooxy)methylidene]disodium cannot be used in making high performance gun propellants or high performance rockets fuels due to detonation upon ignition; however, μ-[nitro(nitrooxy)methylidene]disodium does show potential use as a primer in bullet casings.

Safety Info: Use caution and wear gloves and eye protection when handling dinitrogen pentoxide and avoid contact with combustible materials. Use maximum ventilation and use caution when handling carbon monoxide, which is highly toxic. Use proper ventilation and extinguish all sources of ignition when handling ether, which is highly volatile and forms explosive mixtures with air—also, before using ether conduct a peroxide test as heating ether containing peroxides can explode. Avoid inhalation of ether vapors, as ether is a narcotic in high concentrations. Extinguish all sources of ignition and use proper ventilation when handling hexane, which is highly flammable. Use extreme care when handling metallic sodium as metallic sodium is highly reactive and will explode in contact with water. Wear gloves when handling metallic sodium.

Materials:

1. 5 grams of platinum gauze	7. 575 milliliters of methylene chloride
2. 127 grams of dinitrogen pentoxide	8. 61 grams of sulfur dichloride
3. Carbon dioxide from tank	9. 500 milliliters of diethyl ether
4. 33 grams of carbon monoxide	10. 1500 milliliters of hexane
5. 500 milliliters of ethyl acetate	11. 44 grams of dry metallic sodium
6. 3250 milliliters of 95% ethyl alcohol	12. 2500 milliliters of dry glycerol

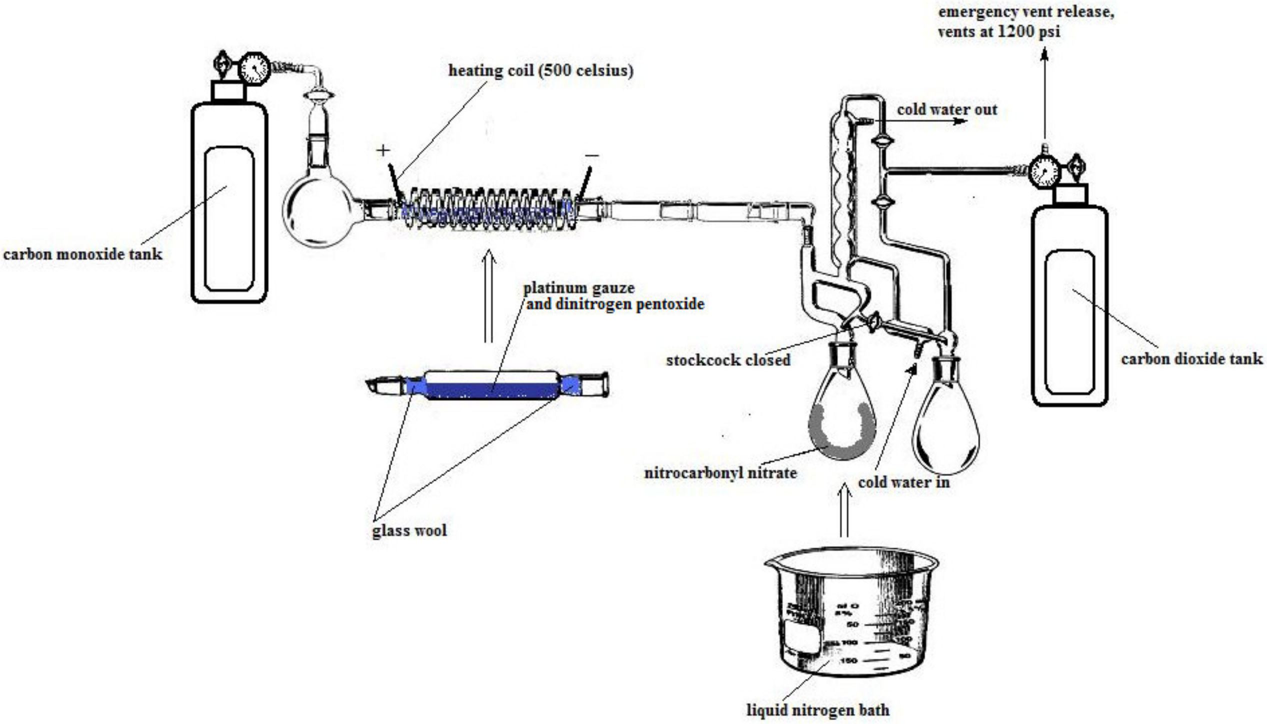
Reaction Equation:



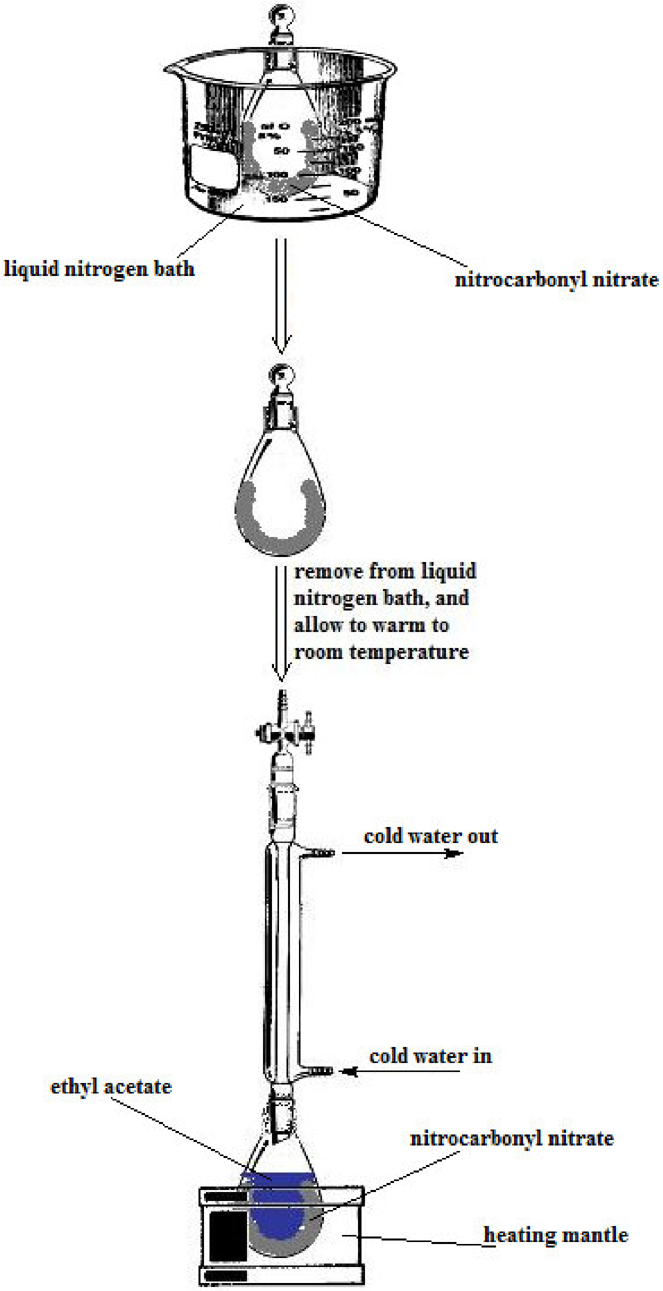
Preparation:

Step 1: preparation of nitrocarbonyl nitrate

Assemble a special apparatus as similar to the one illustrated below, and then place 5 grams of platinum gauze into the glass condenser as illustrated below. Thereafter, place 127 grams of dinitrogen pentoxide into the glass condenser as illustrated below, and then seal both ends of the glass condenser with glass wool. Note: shake the glass condenser tube so that the dinitrogen pentoxide is evenly distributed over the surface. Thereafter, seal the apparatus, and then begin a carbon dioxide gas purge under a flow rate of 500 mg per/minute and bring the pressure inside the apparatus to around 1000 psi. When the pressure inside the apparatus reaches around 1000 psi, turn on the heating coil wrapped around the glass condenser tube as illustrated, and heat this glass condenser tube containing the dinitrogen pentoxide/platinum gauze to 500 Celsius. Once the temperature reaches 500 Celsius, begin to purge the apparatus with carbon monoxide at a flow rate of 50 mg per/minute. Continue to purge the apparatus with carbon dioxide at the aforementioned flow rate, and continue to purge the apparatus with carbon monoxide gas while heating the glass condenser tube containing the dinitrogen pentoxide/platinum to 500 Celsius and maintaining the pressure inside the apparatus at 1000 psi, until approximately 33 grams of carbon monoxide gas has been added. Once all the carbon monoxide gas has been passed into the apparatus, turn-off the valves to the gasses sources, release the pressure release gauge to bring the pressure inside the apparatus to standard atmospheric pressure, and turn off the heating coil around the glass condenser tube, and allow this glass apparatus tube to cool to room temperature. Note: during the reaction process, the solid desired product of nitrocarbonyl nitrate will form, and will volatize and be carried over to the receiver flask submerged in liquid nitrogen as illustrated below, where-by it will crystallize to a beautiful crystalline mass. Now, once the glass condenser tube has cooled to room temperature, disassemble the apparatus if desired, and then remove the receiver flask containing the desired product of nitrocarbonyl nitrate from the liquid nitrogen bath, and allow it to warm to room temperature. Now, once the nitrocarbonyl nitrate has cooled to room temperature, assemble the apparatus as illustrated in the second illustration, and then pour in 500 milliliters of ethyl acetate, and then turn on the heating mantle and reflux the mixture at 80 Celsius for 4 hours. After 4 hours, turn off the heating mantle, and allow the refluxed mixture to cool to room temperature. Finally, once the nitrocarbonyl nitrate/ethyl acetate mixture has cooled to room temperature, pour the mixture into a suitable sized beaker, and then pour in 500 milliliters of 95% ethyl alcohol, and then stir this new mixture for 1 hour at room temperature. After stirring for 1 hour, filter-off the desired product of nitrocarbonyl nitrate using suction filtration, wash with three 250-milliliter portions of fresh 95% ethyl alcohol, followed by washing with three 100-milliliter portions of hot water, and then suction dry as best as possible. Now, before using this washed suction dried product of nitrocarbonyl nitrate, place it into a dessicator over phosphorus pentoxide for 1 week before using in step 2.



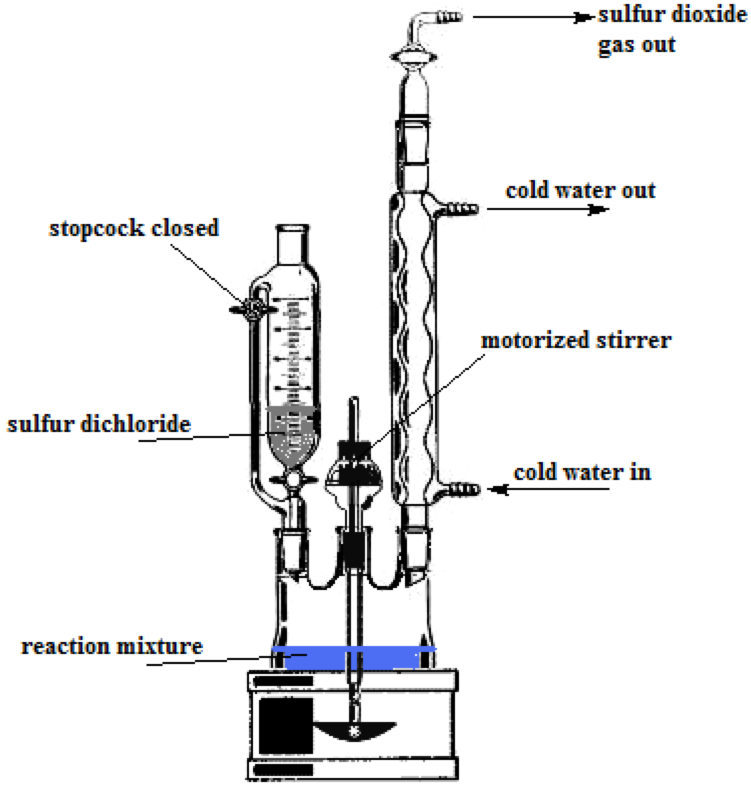
All stainless steal apparatus for preparing nitrocarbonyl nitrate.



Setup for refluxing the nitrocarbonyl nitrate with ethyl acetate to dislodge the nitrocarbonyl nitrate and partially dissolve it.

Step 2: preparation of dichloro(nitro)methyl nitrate

Into an apparatus as illustrated below, place the nitrocarbonyl nitrate obtained in step 1, followed by 575 milliliters of methylene chloride. Immediately thereafter, begin to stir the reaction mixture, and at the same time, pour in 61 grams of sulfur dichloride into the addition funnel. Then, turn on the heating mantle to 40 Celsius, and begin a reflux of the methylene chloride. Once the reaction mixture begins to reflux, slowly add in, drop by drop, the sulfur dichloride into the reaction mixture while vigorously stirring the reaction mixture and maintaining its temperature under reflux at 40 Celsius. Note: use proper ventilation or neutralize the sulfur dioxide by-product that will form during the reaction. After the all the sulfur dichloride has been added, continue to stir the reaction mixture under reflux at 40 Celsius for 2 hours. After 2 hours, turn off the heating mantle and allow the reaction mixture to cool to room temperature. Note: during the cool down period continue to vigorously stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the reaction mixture into a suitable sized beaker, and then pour in 500 milliliters of diethyl ether and then stir this new mixture for 4 hours at room temperature. After stirring for 4 hours, stop stirring, and then filter-off the insoluble desired product of dichloro(nitro)methyl nitrate using suction filtration, and then wash with three 500-milliliter portions of 95% ethyl alcohol, and then suction dry as best as possible, and save the this suction dried product of dichloro(nitro)methyl nitrate for step 3.

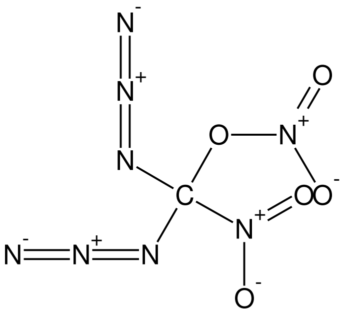
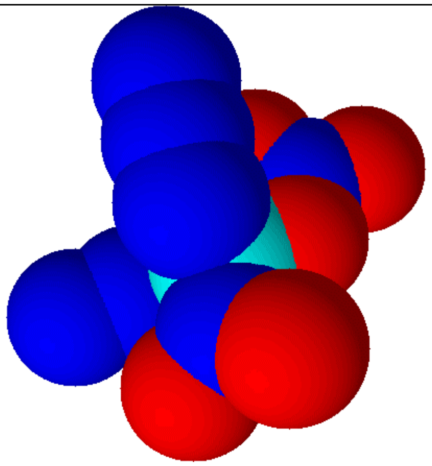


Setup for the chlorination of nitrocarbonyl nitrate.

Step 3: preparation of μ-[nitro(nitrooxy)methylidene]disodium

Into a two-neck flask sitting inside a heating mantle and equipped with motorized stirrer, place the product of dichloro(nitro)methyl nitrate obtained in step 2 into this two-neck flask, and then pour in 750 milliliters of hexane. Thereafter, slowly, and in very small pieces at a time, add in 44 grams of dry metallic sodium over a period of about 1 hour, while rapidly stirring the reaction mixture. Once all the metallic sodium has been added, place a reflux condenser onto the 2-neck flask, and then turn on the heating mantle and then reflux the reaction mixture at 80 Celsius for 4 hours with constant stirring. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during this cool down period. Now, once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a large dry beaker, and then add in 2500 milliliters of dry glycerol, and then stir this new mixture for 6 hours at room temperature. Note: during this time the sodium chloride by-product will dissolve in the glycerol leaving behind the insoluble desired product of μ-[nitro(nitrooxy)methylidene]disodium. Finely, after stirring the mixture for 6 hours, filter-off the insoluble desired product of μ-[nitro(nitrooxy)methylidene]disodium using suction filtration, and then wash with three 250-milliliter portions of dry hexane, and then suction dry as best as possible. Then, place this filtered-off suction dried product into a dessicator over phosphorus pentoxide for 1 week before using. Note: after 1 week, keep this desired product of μ-[nitro(nitrooxy)methylidene]disodium in a dessicator until use.

Theoretical Preparation 47: diazido(nitro)methyl nitrate; DANMN; Nitro methoxy nitrate diazide; Nitroazide methyl nitrate; KL-90

 N N N C O N O O N O O N N N diazido(nitro)methyl nitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = CN_8O_5
Formula Weight = 204.0613
Composition = C(5.89%) N(54.91%) O(39.20%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 203.999165 Da
Nominal Mass = 204 Da
Average Mass = 204.0613 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7345; estimated using laser ray spectroscopy	Non-flammable; will not burn even when heated. Amazingly has little reaction with reducing agents, but mixtures with powdered metals mainly powdered aluminum and magnesium will ignite and burn moderately when touched by a flame.	Remarkably very stable. Stable towards heat, shock, percussion, pressure, and friction. A secondary explosive. Requires blasting cap or detonator for initiation.	Virtually non-reactive. Does not react with halogens, or strong oxidizing agents; however explodes when heated with fluorine. Forms explosive mixtures with alkali metals when ignited by a flame.	White crystalline cubic-like crystals, whiter powder, granules, or flakes. Melting point: 134 Celsius; Boiling point: decomposes when heated to above 210 Celsius. Warning! This compound is poisonous. Insoluble in water,

				and most organic solvents. Practically insoluble in liquid ammonia.

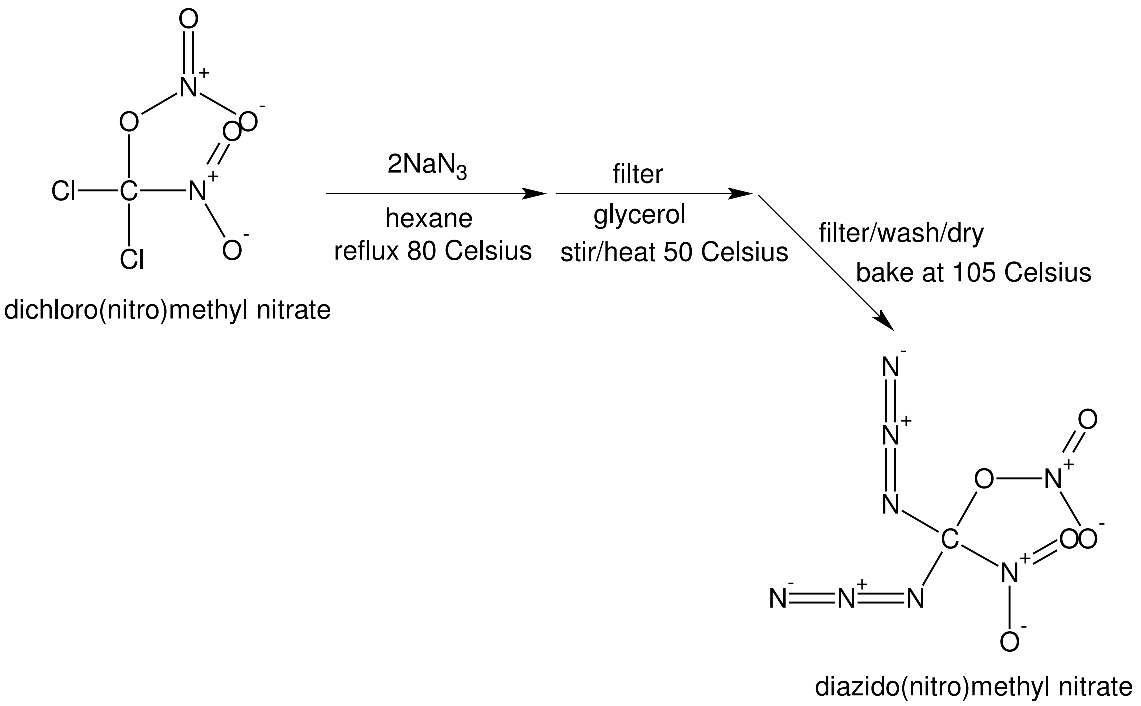
Estimated Compound summary: diazido(nitro)methyl nitrate is a very interesting nitro, nitrate, and azide containing explosive compound with remarkable physical properties. It is very stable, and can be melted down and poured into molds for forming anything from shape charges for anti-tank operations, or demolition operations for commercial and military purposes. Because of its high gas ratio upon detonation diazido(nitro)methyl nitrate has tremendous pressure-heaving action making it perfect for blasting very hard rock for commercial and military operations. The fact diazido(nitro)methyl nitrate is non-flammable makes it a very interesting and useful “safety” explosive for various operations requiring a significant blasting cap or detonator for initiation; nonetheless, it cannot be used to make rocket or gun propellants due to its lack of flammability and reactivity when mixed with reducing agents; however, even though diazido(nitro)methyl nitrate is relatively non-reactive when mixed with reducing agents and ignited, it is however capable of forming dangerously explosive compositions when mixed with alkali metals such as metallic sodium or potassium, and cured with epoxy or a high energy plasticizer and cured—these compositions cannot be used to make high performance rocket propellants but can be used to make modified non nitrocellulose or nitroglycerine containing gun propellants. These metallic sodium or potassium compositions can also be used in making pyrotechnic compositions (of limited use) for use in making fireworks, flares, and a wide variety of pyrotechnic applications. diazido(nitro)methyl nitrate compositions containing powdered aluminum or magnesium can be used in making brilliant white glowing tracer compositions for bullets, shells, and rockets, and also in making very brilliant flares for ground or aerial use. diazido(nitro)methyl nitrate in the molten state is a smooth flowing liquid that can be casted and molded for making a variety of explosive munitions such as mortar shells, artillery shells, anti-tank shaped charge warheads, missile warheads and because it is stable towards most chemical attacks and is remarkably resistant to water, it can be used in making land mines and torpedoes.

Safety Info: Warning! Use caution when handling chloro(nitro)methyl nitrate, which is a vesicant. Extinguish all flames and sources of ignition when handling hexane, which is highly volatile and flammable. Wear gloves when handling sodium azide, and avoid skin contact and ingestion, as sodium azide is highly toxic

Materials:

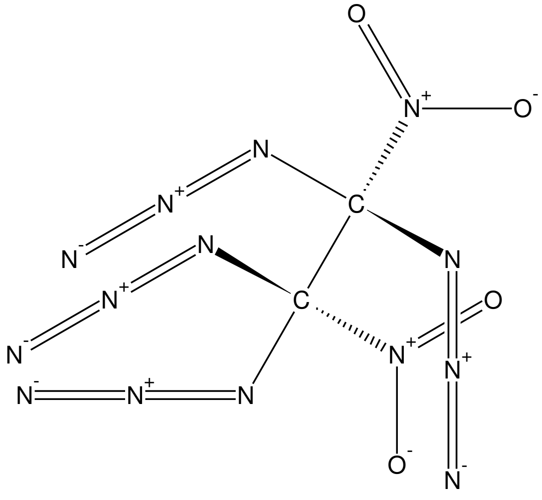
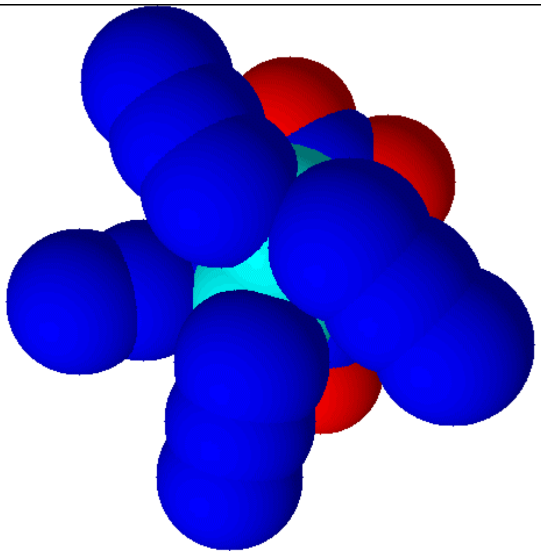
1. 102 grams of chloro(nitro)methyl nitrate (obtained in theoretical preparation 46, step 2)	4. 900 milliliters of 95% ethyl alcohol
2. 500 milliliters of hexane	5. 70 grams of sodium azide
3. 1500 milliliters of glycerol	

Reaction Equation:



Preparation: Into a three-neck flask sitting inside a heating mantle and equipped with motorized stirrer, reflux condenser, and thermometer, place 102 grams of chloro(nitro)methyl nitrate obtained in theoretical preparation 46, step 2, and then pour in 500 milliliters of hexane. Then add in all at once, 70 grams of sodium azide, and then turn on the heating mantle and then reflux the reaction mixture at 80 Celsius for 6 hours with constant stirring. After 6 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to rapidly stir the reaction mixture during this cool down period. Now, once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter-off all the insoluble material using suction filtration, and suction dry as best as possible. Note: this suction dried mass will consist of the desired product and the by-product sodium chloride. Thereafter, place this suction dried mass into a suitable sized beaker sitting on top of a heating mantle, and equipped with motorized stirrer, and then add in 1500 milliliters of glycerol, then heat this beaker containing the said ingredients at 50 Celsius utilizing the motorized stirrer and vigorously stir and heat this mixture for 4 hours. Note: during this time period the by-product sodium chloride will dissolve into the glycerol, leaving the insoluble desired product behind. After stirring and heating at 50 Celsius for 4 hours, filter-off the insoluble desired product of diazido(nitro)methyl nitrate using suction filtration, and then wash with three 300-milliliter portions of 95% ethyl alcohol, and then suction dry as best as possible. Then, place this filtered-off suction dried product into an oven, and bake at 105 Celsius for 12 hours. After 12 hours, turn off the oven, and allow the dried desired product of diazido(nitro)methyl nitrate to cool to room temperature. Once it has cooled to room temperature, remove the dried desired product and store in an amber glass bottle in a cool dry place until use.

Theoretical Preparation 48: 1,1,2,2-tetraazido-1,2-dinitroethane; TADNE; E^t-dinitro tetraazide; dinitroethane poly azide;

 1,1,2,2-tetraazido-1,2-dinitroethane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_2N_{14}O_4$
Formula Weight = 284.1128
Composition = C(8.45%) N(69.02%) O(22.53%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 284.022695 Da
Nominal Mass = 284 Da
Average Mass = 284.1128 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6987	Non-flammable; will not burn even when ignited in excess oxygen; however will ignite when heated to over 200 Celsius whereby it sputters and spits, forming nitrous oxide, nitrogen oxides, and carbon monoxide.	Very stable. Non-sensitive to shock, heat, percussion, pressure, and friction. A small sample will not even detonate when struck with a heavy hammer. A secondary explosive. Requires significant	Virtually non-reactive. Does not react with strong oxidizers, acids, bases, or reducing agents even when heated.	Colorless to white crystalline mass, granules, or powder. Melting point: 167 Celsius; boiling point: decomposes when heated to 238 Celsius. Insoluble in water, and all

		blasting cap or detonator for initiation.		known solvents.

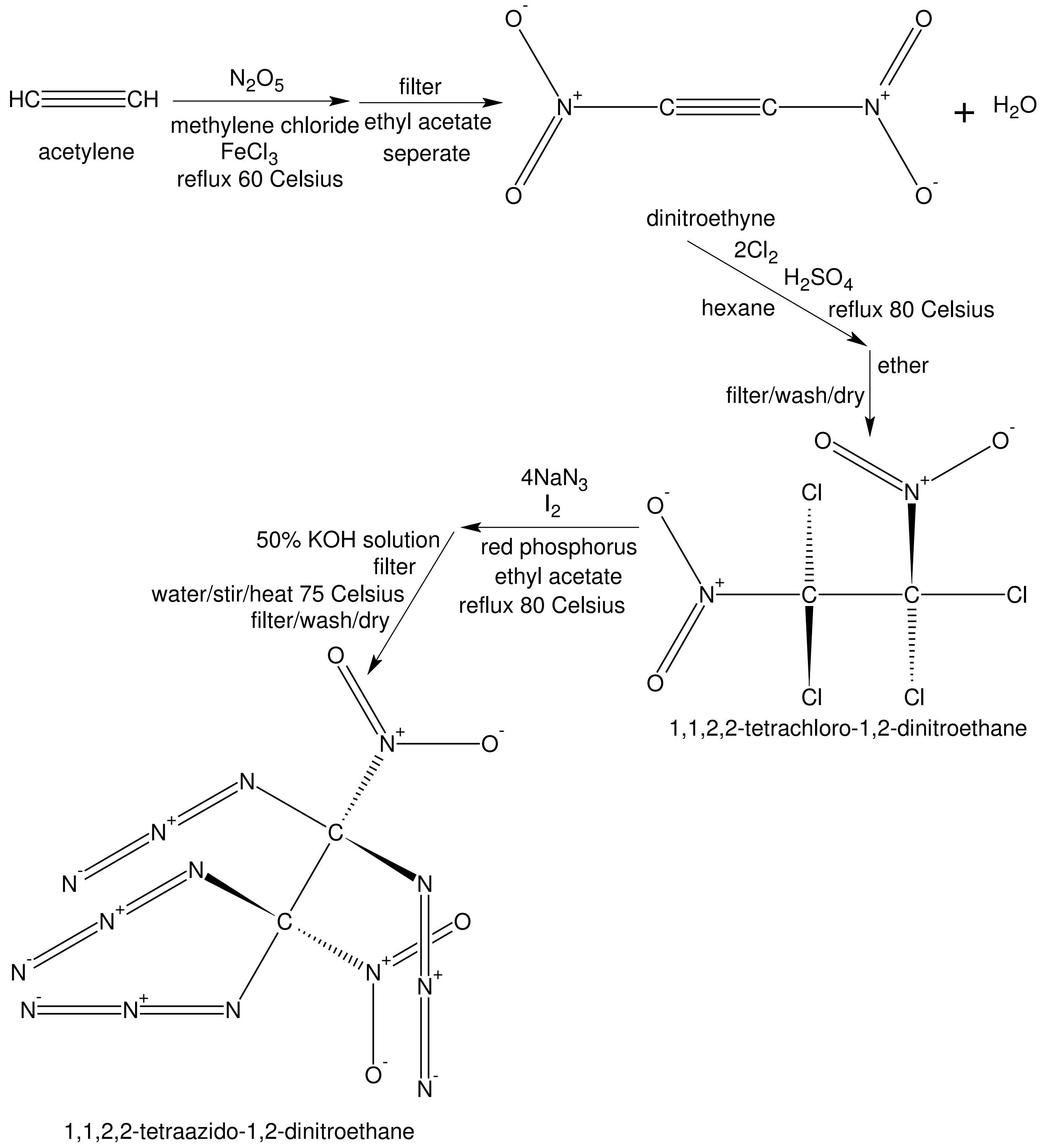
Estimated Compound summary: 1,1,2,2-tetraazido-1,2-dinitroethane is very similar to diazido(nitro)methyl nitrate, and is a very interesting nitro, and azide containing explosive compound with remarkable physical properties. It is very stable, and can be melted down and poured into molds for forming anything from shape charges for anti-tank operations, or demolition operations for commercial and military purposes. Because of its high gas ratio upon detonation 1,1,2,2-tetraazido-1,2-dinitroethane has tremendous pressure-heaving action making it perfect for blasting very hard rock for commercial and military operations. The fact 1,1,2,2-tetraazido-1,2-dinitroethane is non-flammable makes it a very interesting and useful "safety" explosive for various operations requiring a significant blasting cap or detonator for initiation; nonetheless, like its predecessor diazido(nitro)methyl nitrate, it cannot be used to make rocket or gun propellants due to its lack of flammability and reactivity when mixed with reducing agents. Also, unlike diazido(nitro)methyl nitrate, 1,1,2,2-tetraazido-1,2-dinitroethane is almost virtually non-reactive when mixed with reducing agents and ignited. Even when melted with alkali metals such as metallic sodium or potassium, it forms very stable mixtures that only slowly react with water without any undo danger, and hence these mixtures have practically no use. 1,1,2,2-tetraazido-1,2-dinitroethane has no use in making pyrotechnic compositions; However, like diazido(nitro)methyl nitrate, 1,1,2,2-tetraazido-1,2-dinitroethane compositions containing powdered aluminum or magnesium can be used in making brilliant white glowing tracer compositions for bullets, shells, and rockets, and also in making very brilliant flares for ground or aerial use. Like diazido(nitro)methyl nitrate, 1,1,2,2-tetraazido-1,2-dinitroethane in the molten state is a smooth flowing liquid that can be casted and molded for making a variety of explosive munitions such as mortar shells, artillery shells, anti-tank shaped charge warheads, missile warheads and because it is stable towards most chemical attacks and is remarkably resistant to water, it can be used in making land mines and torpedoes. It should be noted, 1,1,2,2-tetraazido-1,2-dinitroethane is insoluble in all known molten explosives such as TNT, PETN, HMX and similar molten explosives.

Safety Info: Use caution when handling dinitrogen pentoxide, and avoid contact with combustible materials. Extinguish all flames when handling acetylene gas, and avoid contact of acetylene with copper compounds. Extinguish all sources of ignition when handling hexane, which is highly volatile. Wear gloves when handling concentrated sulfuric acid, and avoid skin contact. Use maximum ventilation, and avoid inhalation of chlorine, which is toxic. Extinguish all flames when handling diethyl ether, which is highly volatile and forms explosive mixtures with air. Perform the peroxide test before using stored diethyl ether. Wear gloves when handling iodine, and avoid skin contact and ingestion. Wear gloves and use caution when handling sodium azide, which is very toxic. Wear gloves, and avoid skin contact and ingestion when handling concentrated potassium hydroxide solution.

Materials:

1. 2000 milliliters of methylene chloride	8. 401 grams of chlorine gas
2. 403 grams of dinitrogen pentoxide	9. 2400 milliliters of diethyl ether
3. 90 grams of anhydrous ferric chloride	10. 15 grams of double sublimed iodine
4. 97 grams of acetylene gas	11. 50 grams of powdered red phosphorus
5. 4000 milliliters of ethyl acetate	12. 735 grams of sodium azide
6. 1500 milliliters of dry hexane	13. 350 milliliters of a 50% potassium hydroxide solution
7. 5 milliliters of concentrated sulfuric acid	

Reaction Equation:

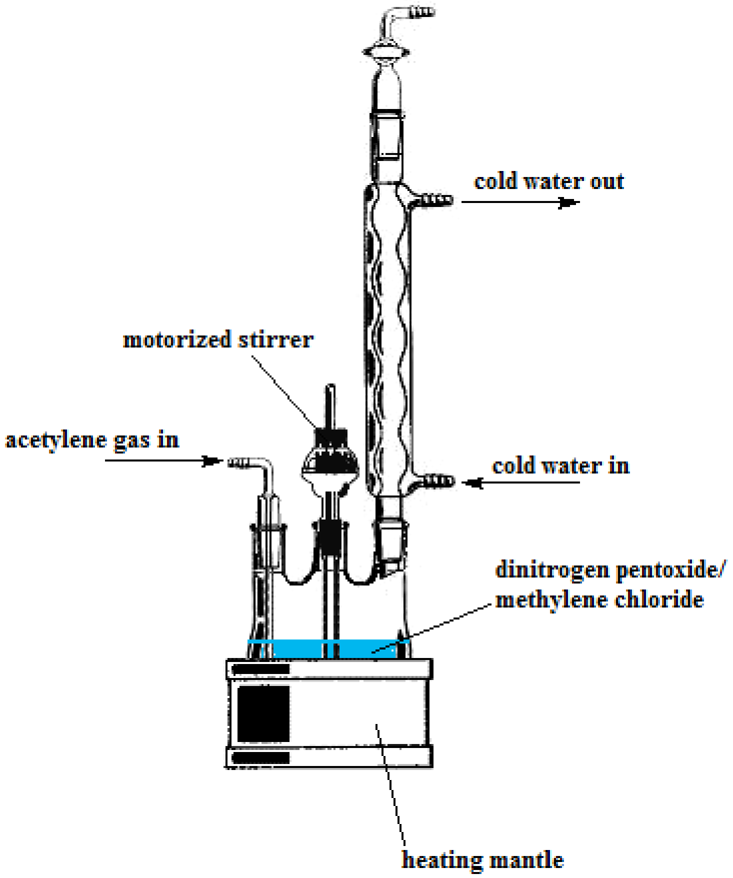


Preparation:

Step 1: preparation of dinitroethyne (dinitro acetylene)

Note: dinitro acetylene can be used as a liquid explosive, but it is a very dangerous substance to handle. Keep it away from open flames, excessive shock or percussion, from reducing agents, and keep it away from copper and copper compounds—the two latter's form very shock sensitive materials that easily detonate.

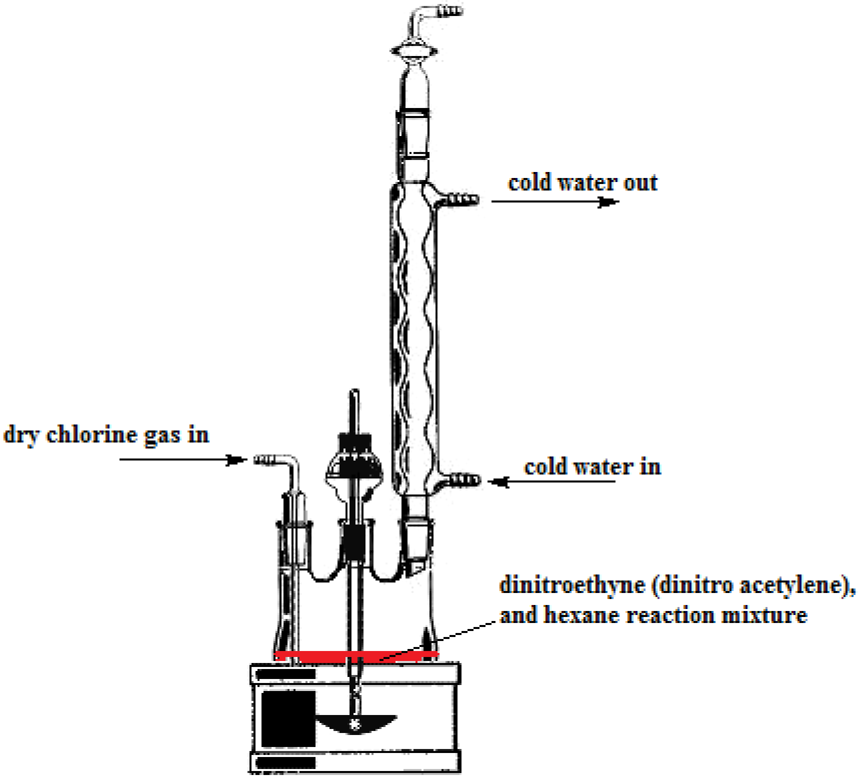
Into an apparatus as the one illustrated below, place 2000 milliliters of methylene chloride containing 403 grams of dinitrogen pentoxide, and then add in 90 grams of anhydrous ferric chloride. Thereafter, turn on the heating mantle and bring the temperature of the reaction mixture to 60 Celsius under vigorous reflux, and turn on the motorized stirrer and begin to stir the reaction mixture vigorously. Once the reaction mixtures temperature reaches 60 Celsius, begin to bubble in acetylene gas at a flow rate of 50 mg per/minute, and continue to reflux the reaction mixture under reflux with constant stirring until 97 grams of acetylene gas has been added. Once all the acetylene gas has been bubbled into the reaction mixture, continue to reflux and vigorously stir the reaction mixture at 60 Celsius for 6 hours. Now, after 6 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, carefully disassemble the apparatus, and then filter the reaction mixture using suction filtration to recover insoluble impurities (mostly hydrated ferric chloride). Thereafter, take the filtered reaction mixture, and pour it into a pre-chilled beaker (placed into a freezer prior to use) and then pour in 1500 milliliters of ethyl acetate, and then stir this new mixture for 2 hours. Note: when the ethyl acetate is added, all of the desired liquid product of dinitro acetylene will separate out as a viscous liquid forming an upper layer, with the methylene chloride/ethyl acetate mixture forming the bottom layer. Now, either decant this upper layer into a clean beaker, or use a large separatory funnel to drain-off the bottom methylene chloride/ethyl acetate mixture to collect the desired liquid product of dinitro acetylene. Either way, once the upper liquid layer of dinitro acetylene has been recovered, save it an amber glass bottle in a refrigerator until use for step 2.



Nitration of acetylene.

Step 2: preparation of 1,1,2,2-tetrachloro-1,2-dinitroethane

Into an apparatus as similar to the one illustrated below, pour in the dinitroethyne (dinitro acetylene) product obtained in step1, and then pour in 1500 milliliters of dry hexane, followed by 5 milliliters of concentrated sulfuric acid. Thereafter assemble the apparatus, and then turn on the heating mantle and bring the reaction mixture to reflux at 80 Celsius. Once the reaction mixture has reached 80 Celsius, begin to bubble into the reaction mixture dry chlorine gas at a flow rate of 120 mg per/minute while continuing to reflux the reaction mixture under vigorous stirring. Continue to bubble into the reaction mixture dry chlorine gas under vigorous stirring and refluxing at 80 Celsius until 401 grams of chlorine gas has been added. Once 401 grams of dry chlorine gas has been added, turn off the gas source, and then continue to reflux the reaction mixture at 80 Celsius under vigorous stirring for 3 hours. After 3 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Once it has, disassemble the apparatus, and then pour the entire reaction mixture into a large pre-chilled beaker (chilled in a freezer prior to use), and then pour in 1500 milliliters of diethyl ether, and then stir this new mixture for about 1 hour. After 1 hour, filter-off the insoluble desired product of 1,1,2,2-tetrachloro-1,2-dinitroethane using suction filtration, and then wash with three 300-milliliter portions of fresh diethyl ether, and then suction dry as best as possible. Thereafter, save this suction dried product of 1,1,2,2-tetrachloro-1,2-dinitroethane for step 3.

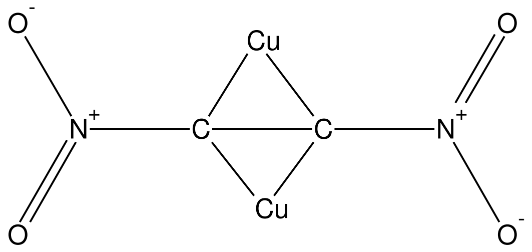

cold water out
dry chlorine gas in
cold water in
dinitroethyne (dinitro acetylene), and hexane reaction mixture

Setup for the chlorination of dinitroethyne (dinitro acetylene).

Step 3: preparation of 1,1,2,2-tetraazido-1,2-dinitroethane

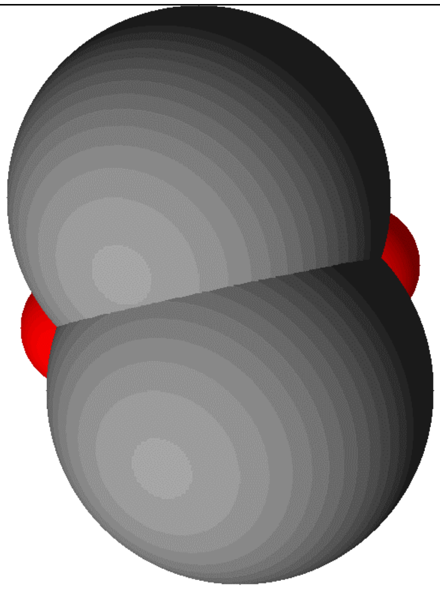
Into a large 3-neck flask sitting inside a round bottom heating mantle and equipped with motorized stirrer, reflux condenser, and thermometer, place into this 3-neck flask the product obtained in step 2 followed by 2500 milliliters of dry ethyl acetate, followed by 15 grams of double sublimed iodine, followed by 50 grams of powdered red phosphorus, and then followed by 735 grams of sodium azide. Thereafter assemble the apparatus, and then turn on the heating mantle, and heat the reaction mixture to 80 Celsius under reflux. When the reaction mixtures temperature reaches 80 Celsius, reflux the reaction mixture under rapid stirring for 8 hours. After 8 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to rapidly and vigorously stir the reaction mixture. Once this reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a large beaker, and then add in 350 milliliters of a 50% potassium hydroxide solution made by adding and dissolving 150 grams of potassium hydroxide into 150 milliliters of water. Note: dissolving potassium hydroxide in water produces a large amount of heat so prepare this solution before starting this procedure to allow the potassium hydroxide solution time to cool before use. After adding in the 50% potassium hydroxide solution to the reaction mixture in the beaker, stir this new mixture for 4 hours. Now, after stirring for 4 hours, filter this mixture to recover the insoluble desired product of 1,1,2,2-tetraazido-1,2-dinitroethane (other insoluble products will remain mixed with the desired product), using suction filtration, and then suction dry as best as possible. Finally, take the filtered-off mass containing the desired product and by-products (the filter cake), and place it into a large clean beaker sitting on top of a hot plate, and then add in 3000 milliliters of tap water, and then turn on the hot plate and heat the contents in the beaker to 75 Celsius with stirring (use a motorized stirrer, not a magnetic stirrer). When the temperature of the mixture reaches 75 Celsius, continue to rapidly stir it while heating it to 75 Celsius for 4 hours. Note: during this heating period, all the by-products will dissolve in the water leaving behind a refined almost pure desired product of 1,1,2,2-tetraazido-1,2-dinitroethane. Now, after heating and stirring for 4 hours, turn off the hot plate and turn off the stirring device, and then filter the entire mixture (while still hot) using suction filtration to recover the insoluble desired product of 1,1,2,2-tetraazido-1,2-dinitroethane. Thereafter, wash this recovered filtered-off desired product with six 500-milliliter portions of hot tap water, and then suction dry as best as possible. Afterwards, place this suction dried desired product into an oven and bake at 110 Celsius for 12 hours. After 12 hours, turn off the oven, and then allow the dried desired product of 1,1,2,2-tetraazido-1,2-dinitroethane to cool to room temperature. Once it has cooled to room temperature remove the dried desired product from the oven, and store in a glass jar in a cool dry place until use.

Theoretical Preparation 49: 1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane; Dinitro copper-II-acetylide; DNC2A; Dinitro acetylene copper salt



1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane

Expanded structure



3D structure

Molecular data:

Molecular Formula = $C_2Cu_2N_2O_4$
Formula Weight = 243.1254972
Composition = C(9.88%) Cu(52.27%) N(11.52%) O(26.32%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 241.846106 Da
Nominal Mass = 242 Da
Average Mass = 243.1255 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
4578	Non-flammable; explodes when heated to above 120 Celsius. Mixtures with reducing agents explode violently when struck, scratched, or heated.	Very sensitive to shock, heat, friction, and percussion; handle with extreme care. A primary explosive.	Is slowly oxidized by air, and is very slowly hydrolyzed by water. Explodes in contact with the halogens, ozone, sulfur trioxide, and hydrogen sulfide.	Brick-red, amorphous powder. Melting point: explodes when heated to 121 Celsius. Boiling point: N/A; insoluble in water

				and all known solvents.

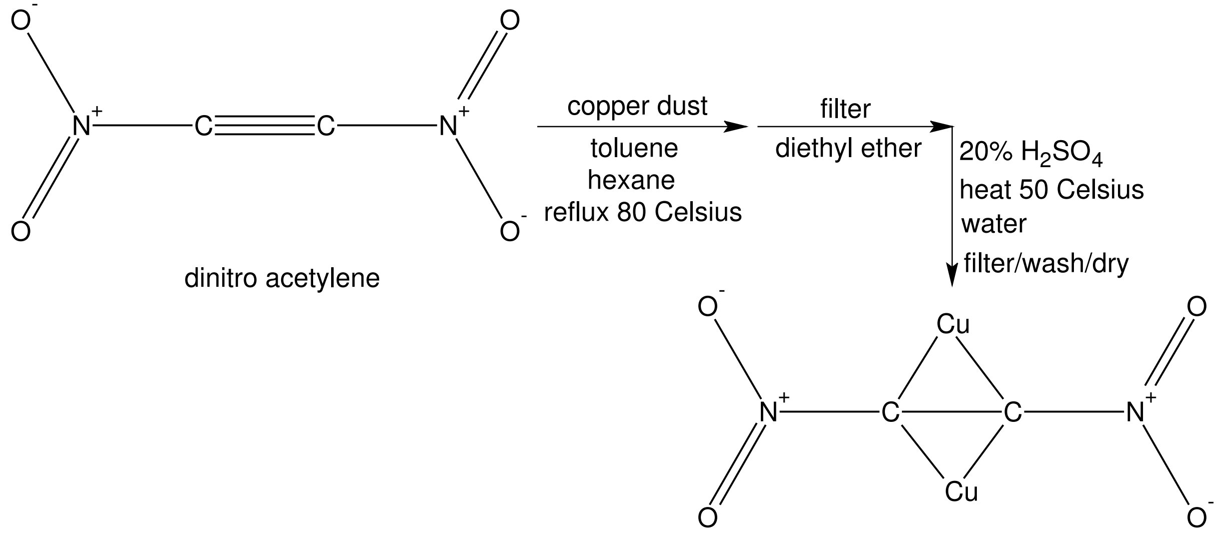
Estimated Compound summary: 1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane is a unique cyclic copper acetylene explosive that is very similar in nature to mercury fulminate, but it is less sensitive then mercury fulminate. It can be used as a substitute for lead azide in blasting caps and detonators, and it does not need to be diluted with inert fillers. The explosive is slowly hydrolyzed by water forming complex hydrated copper acetylides, and loosing its explosiveness—hence it should only be used in blasting caps and detonators when sealed water and airtight to protect it from decomposition. One draw back to this explosive is that it produces carbon monoxide when detonated, and hence should not be used in large quantities in enclosed areas mainly in mining operations. 1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane may not be the primary explosive of choice, but it can be used in blasting caps and detonators for use in mortar shell fuses, artillery shell fuses, and fuses for low velocity munitions; however, 1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane should not be used in fuses for high velocity projectiles mainly tank fired projectiles or fuses for rockets due to its tendency to detonate due to the sudden shock of hyper acceleration. 1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane shows remarkable ability for use in primers for bullets when it is in small grain sizes of 0.5 to 1.0 millimeters (made by slurring it with water then drying, and then crushing followed by sieving). 1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane can also be used in making fireworks of the "fun snap" type using small grains of 1 to 1.5 millimeters and wrapped in a small paper bag with small sand grains—when these little paper bags are thrown onto a hard surface, they make a loud popping sound.

Safety Info: Use extreme care when handling dinitro acetylene, which is sensitive to shock, heat, and percussion. Use proper ventilation when handling toluene, and avoid inhalation of the vapors, as toluene is a known carcinogen. Extinguish all flames, and use proper ventilation when handling hexane, which is highly flammable. Copper dust is flammable so use care. Use maximum ventilation, and avoid inhalation of the vapors when handling diethyl ether, which is a narcotic in high concentrations, and forms explosive mixture with air. Note: always use fresh ether, and perform the peroxide test for ether that has been in storage for prolonged storage.

Materials:

1. 147 grams of dinitro acetylene (prepared in theoretical preparation 48, step 1)	4. 161 grams of copper dust
2. 500 milliliters of toluene	5. 750 milliliters of a 20% sulfuric acid solution
3. 500 milliliters of hexane	6. 750 milliliters of fresh diethyl ether

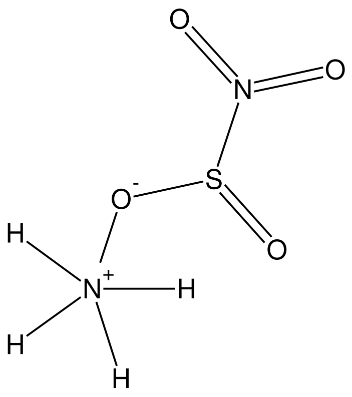
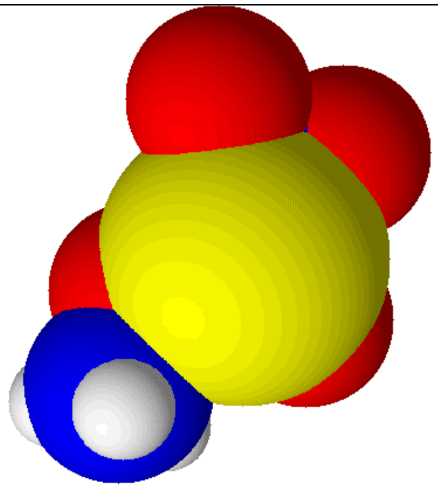
Reaction Equation:



1,3-dinitro-2,4-dicupricbicyclo[1.1.0]ethane

Preparation: Into a suitable sized reflux apparatus equipped with cold water condenser, motorized stirrer, and thermometer, pour in 147 grams of dinitro acetylene, an then quickly add in 500 milliliters of toluene, followed by 500 milliliters of hexane, and then add in all at once, 161 grams of copper dust. Thereafter, seal the apparatus, and turn on the heating mantle, and begin a gentle reflux at 80 Celsius with constant stirring. Continue to reflux the reaction mixture at 80 Celsius with moderate stirring for 8 hours. After 8 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to moderately stir the reaction mixture. Once the reaction mixture has cooled to room temperature, filter the reaction mixture using suction filtration to remove the insoluble desired product. Once the insoluble desired product has been collected on the Buchner funnel, wash this filtered-off desired product with three 250-milliliter portions of fresh diethyl ether, and then suction dry as best as possible. Now, remove the washed filtered-off desired product from the Buchner funnel and place it into a suitable sized beaker, and then add in 750 milliliters of a 20% sulfuric acid solution, and then place the beaker containing these ingredients onto a hot plate and heat to 50 Celsius with stirring. Note: use a motorized stirrer not a magnetic stirrer. Thereafter, continue to stir this new reaction mixture for 3 hours at 50 Celsius. After 3 hours, turn off the heating mantle and allow this new reaction mixture to cool to room temperature. Note: continue to stir this new reaction mixture during the cool down period. Finally, pour in 500 milliliters of hot water, and then continue to stir this reaction mixture for 1 hour. After 1 hour, filter-off the insoluble desired product using suction filtration, wash with twelve 500-milliliter portions of warm water, and then suction dry as best as possible. Then, remove the washed filtered-off desired product from the Buchner funnel, and place it into a dessicator over phosphorus pentoxide for 1 week. After 1 week, the desired product will be dry and ready for use. For prolonged storage, keep it in the dessicator until use.

Theoretical Preparation 50: ammonium oxo(sulfino)azane oxide; ammonium azane; Azane; Azane-Cl

 ammonium oxo(sulfino)azane oxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $H_4N_2O_4S$
Formula Weight = 128.10776
Composition = H(3.15%) N(21.87%) O(49.96%) S(25.03%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = Not available
Nominal Mass = Not available
Average Mass = Not available

Molecular Information:

Detonation velocity	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

(estimated by monoisotopic mass and index of refraction):				
2995; calculated using Fishers equation.	Non-flammable; forms highly flammable mixtures with reducing agents especially powdered metals—in situ see ammonium nitrate	Non-sensitive to shock, heat, percussion, friction, and pressure. Very stable. A secondary explosive. Requires blasting cap or detonator for initiation.	Relatively un-reactive. Shows know reaction to chlorine, bromine, or iodine. Has no reaction with strong oxidizing agents, but is oxidized by potassium permanganate when heated.	White crystalline mass, powder, or granules. Melting point: 189 Celsius; Boiling point: decomposes when heated above 250 Celsius. Soluble in water, and very soluble in boiling water. Soluble in liquid ammonia, and liquid sulfur dioxide. Also soluble in carbon disulfide. Insoluble in all other solvents.

Estimated Compound summary: Ammonium oxo(sulfino)azane oxide is very similar to ammonium nitrate, but is much more stable then ammonium nitrate, and unlike ammonium nitrate, ammonium oxo(sulfino)azane oxide has a relatively moderate melting point making it very unique, and giving it very useful properties for a large variety of operations. It can be melted down and casted into molds for making a variety of explosive munitions (note: molten ammonium oxo(sulfino)azane oxide is insoluble in other molten explosives such as TNT, RDX, HMX, PETN, ect.,), however, its low detonating velocity does not make it well suitable for making shape charges or many other explosive munitions of military importance. Nonetheless, ammonium oxo(sulfino)azane oxide can be melted, mixed with powdered aluminum and then cured to make a moderate high velocity rocket fuel. Also ammonium oxo(sulfino)azane oxide can also be used in pyrotechnic mixtures with reducing agents for making all sorts of mixtures with multiple uses such as flares, fireworks, Bengal lights, torches, matches, gas generating mixtures, gun propellants, and many other pyrotechnic formulations, many of which, have not been investigated thoroughly. Ammonium oxo(sulfino)azane oxide can also be used in place of ammonium nitrate in pyrotechnic compositions, gun propellants, and rocket fuels whereby the operations of making the said mixtures does not change except ammonium oxo(sulfino)azane oxide is used in place of the ammonium nitrate. Ammonium oxo(sulfino)azane oxide when mixed with a suitable reducing agent and cured with epoxy makes a very powerful gas generating substance that can be used as a propellant for mortars, low velocity tube fired projectiles such as a 40mm grenade launchers, or for propelling any other low velocity device/munition. For use in fireworks, ammonium oxo(sulfino)azane oxide when mixed with potassium permanganate, can be used as a bursting charge for aerial fireworks. One of the most common uses of ammonium oxo(sulfino)azane oxide is in making safety explosives in place of ammonium nitrate for blasting soft rock for mining operations—like ammonium nitrate, ammonium oxo(sulfino)azane oxide can be sprayed directly into bore holes, primed, and then detonated. Also, like ammonium nitrate, ammonium oxo(sulfino)azane oxide forms prills when mixed with small amounts of fuel oil. These mixtures can be used in low velocity explosives for demolition operations in crowded urban environments whereby fragmentation and control of blasting and debris collection can be closely monitored and controlled. Ammonium oxo(sulfino)azane oxide prills can also be used in mining operations either above or below ground.

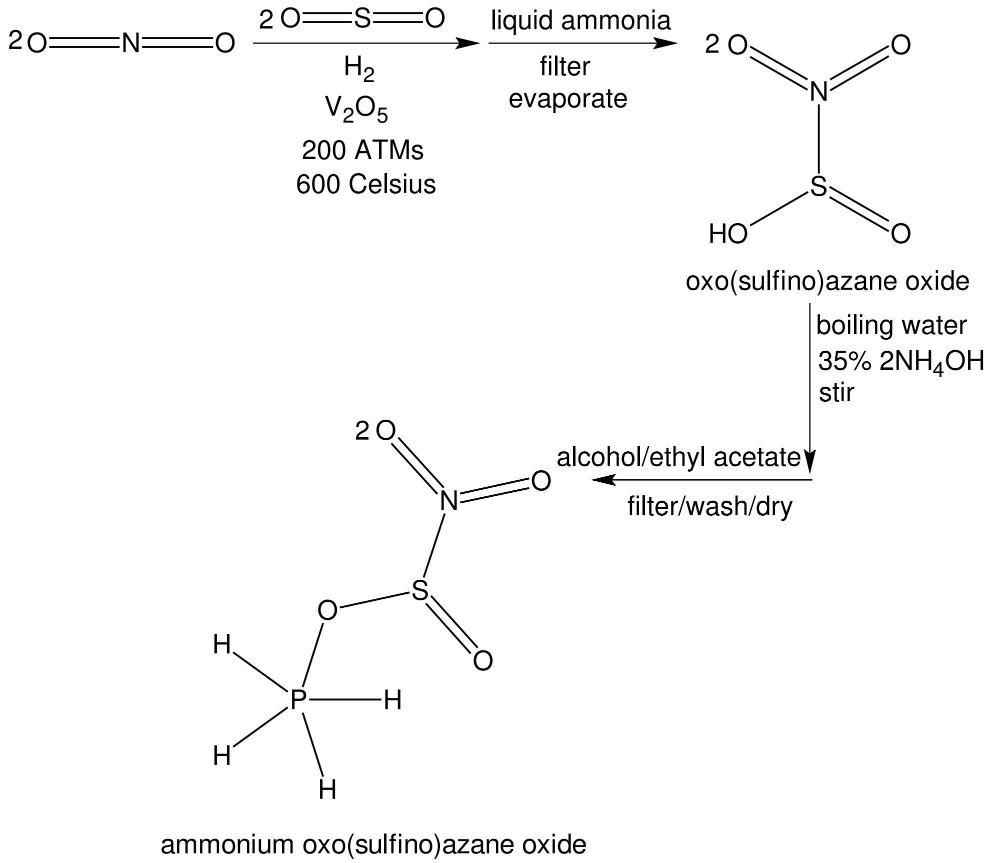
Safety Info: Use maximum ventilation and avoid inhalation of nitrogen dioxide gas, which is very toxic. Use adequate ventilation when handling sulfur dioxide, which is highly irritating to the eyes, nose, and throat. Use care when handling hydrogen gas, which forms explosives mixtures with air. Use great care when handling liquid ammonia and use maximum ventilation and avoid inhalation of the vapors. Ammonia is highly irritating to the mucous membranes. Aqueous ammonia is highly irritating to the mucous membranes so use care. Isopropyl alcohol, ethyl acetate, and hexane are volatile and flammable so take necessary precautions.

Materials:

1. 250 grams of freshly prepared vanadium pentoxide	6. 4 liters of liquid ammonia
2. Argon gas from tank	7. 165 grams of a 35% aqueous ammonia solution
3. 157 grams of nitrogen dioxide gas	8. 500 milliliters of 99% isopropyl alcohol

4. 218 grams of sulfur dioxide gas	9. 500 milliliters of ethyl acetate
5. 3.5 grams of hydrogen gas	10. 1500 milliliters of hexane

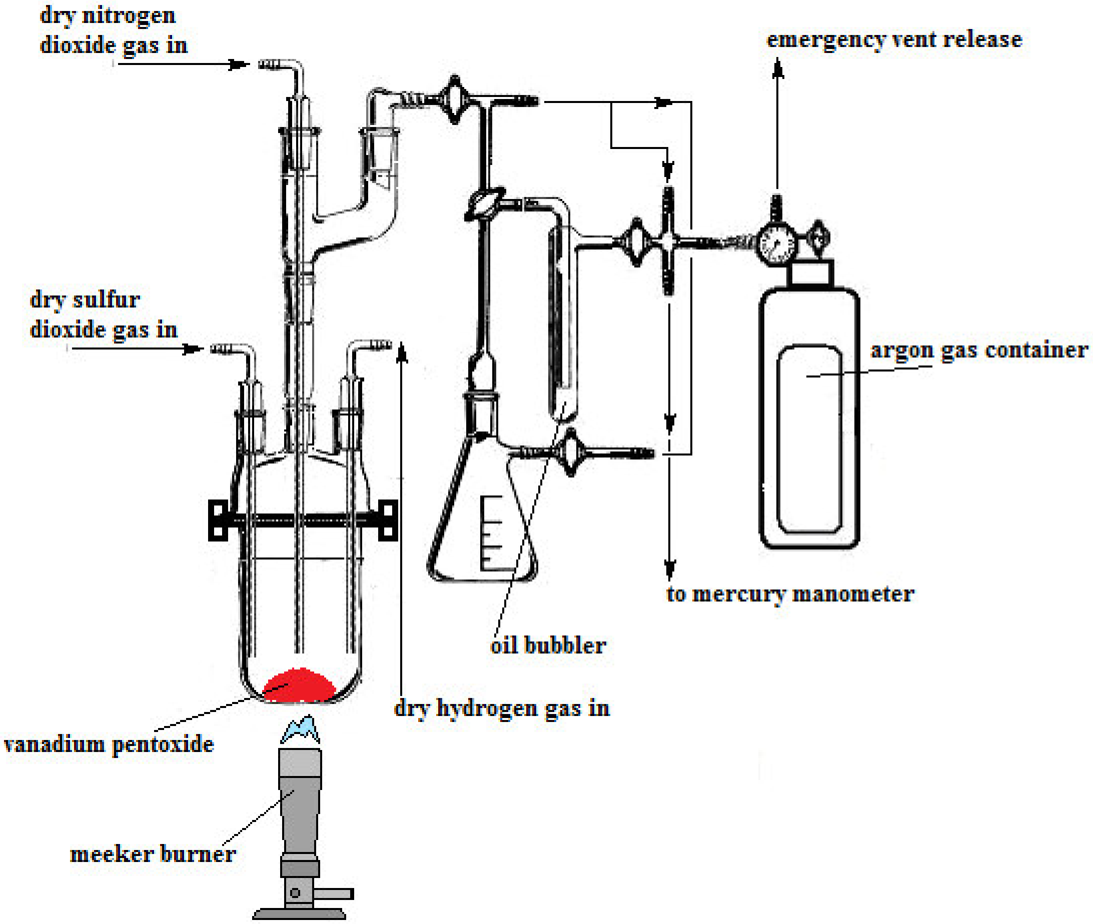
Reaction Equation:



Preparation:

Step 1: preparation of oxo(sulfino)azane oxide

Into an apparatus as illustrated below, place 250 grams of freshly prepared vanadium pentoxide into the reaction vessel as illustrated, and then turn on the argon gas container to 2940 psi. When the pressure inside the apparatus reaches 2940 psi (200 ATMs, turn on the meeker burner, and bring the temperature inside the reaction vessel to 600 Celsius. Once the pressure is at 200 ATMs, and the temperature inside the reaction vessel has reached 600 Celsius, begin to pass in dry nitrogen dioxide gas at a flow rate of 45 mg per/minute, and at the same time begin to pass in dry sulfur dioxide gas at a flow rate of 62 mg per/minute, and at the same time begin to pass in dry hydrogen gas at a flow rate of 9.3 mg per/minute. Now, continue to pass in all three gasses at their respective flow rates until a total of 157 grams of nitrogen dioxide gas has been added, 218 grams of sulfur dioxide gas has been added, and 3.5 grams of hydrogen gas has been added, while maintaining a pressure inside the reaction vessel at 200 ATMs at a temperature of 600 Celsius. Once all three gasses have been added into the reaction vessel at their respective mass, shut off all three gas sources, turn off the meeker burner, and turn off the argon gas purge, and release the emergency vent release to bring the pressure inside the reaction vessel back to standard atmospheric pressure. Once the temperature inside the reaction vessel has cooled to room temperature, disassemble the apparatus, and then take the reaction vessel (removing the three gas inlet adapters) and the reaction vessels lid, and soak and submerge it in a large bath of liquid ammonia to dissolve the desired product of oxo(sulfino)azane oxide which will be stuck to the walls of the reaction vessel and its lid. Note: this operation requires a large amount of liquid ammonia, approximately 4 liters using a deep tall container so that the reaction vessel and its lid can be completely submerged. Then place this liquid ammonia bath containing the submerged reaction vessel and lid, and place it into a sonic vibrating machine to vibrate the reaction vessel and its lid to dislodge the desired product of oxo(sulfino)azane oxide, and allowing it to dissolve in the liquid ammonia. Continue to vibrate the liquid ammonia bath for 4 hours, and after 4 hours, turn off the sonic vibrator machine, and then pluck out the reaction vessel and lid, and place aside. Finally, using a super large Buckner funnel, filter the liquid ammonia mixture using suction filtration to recover the insoluble vanadium pentoxide. Thereafter, pour the filtered liquid ammonia into a shallow pan and allow it to evaporate away. Note: Use proper ventilation when evaporating-off the liquid ammonia. Once the liquid ammonia has been evaporated away and dry residue remains, scrape up the dried residue, which will be the desired product of oxo(sulfino)azane oxide, and save it for step 2.

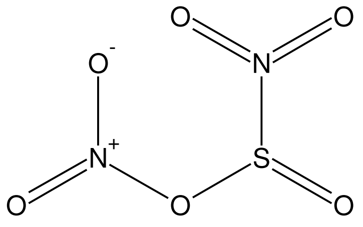


All Nickel setup for preparing oxo(sulfino)azane oxide.

Step 2: preparation of ammonium oxo(sulfino)azane oxide

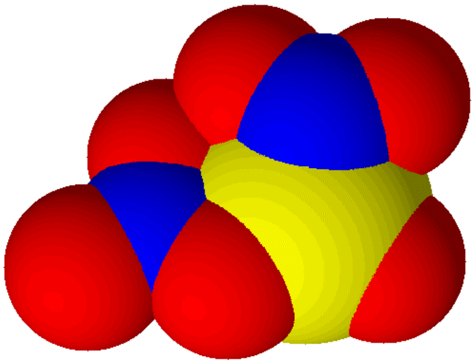
Into a very large beaker sitting on top of a hot plate and equipped with motorized stirrer (not magnetic stirrer), place the dried residue of oxo(sulfino)azane oxide obtained in step 1, and then add in all at once 1500 milliliters of pre heated boiling water, followed by adding in all at once 165 grams of a 35% aqueous ammonia solution, and begin to stir this reaction mixture vigorously for 2 hours. During this 2-hour period, the reaction mixture will slowly cool down. After 2 hours, pour in a solvent mixture obtained by mixing 500 milliliters of 99% isopropyl alcohol with 500 milliliters of ethyl acetate, and then continue to rapidly stir the reaction mixture for 1 hour. After 1 hour, filter-off the precipitated desired product of ammonium oxo(sulfino)azane oxide using suction filtration, and then wash this filtered-off desired product with three 500-milliter portions of hexane, and then suction dry as best as possible. Finely, place the suction dried desired product into an oven and bake at 105 Celsius for 12 hours. After 12 hours, turn off the oven, and allow the dried desired product to cool to room temperature. Once it has, remove the cooled dried desired product from the oven and store it in a dessicator over concentrated sulfuric acid until use.

Theoretical Preparation 51: [(nitrooxy)sulfinyl](oxo)azane oxide; Nitroxy azane; Nitro nitroxy azane; Azane-109



[(nitrooxy)sulfinyl](oxo)azane oxide

Expanded structure



3D structure

Molecular data:

Molecular Formula = N_2O_6S
Formula Weight = 156.0748
Composition = N(17.95%) O(61.51%) S(20.54%)
Molar Refractivity = 24.70 ± 0.3 cm^3
Molar Volume = 69.9 ± 3.0 cm^3
Parachor = 239.8 ± 4.0 cm^3
Index of Refraction = 1.624 ± 0.02
Surface Tension = 138.1 ± 3.0 dyne/cm
Density = 2.230 ± 0.06 g/cm^3
Dielectric Constant = Not available
Polarizability = 9.79 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 155.947706 Da
Nominal Mass = 156 Da
Average Mass = 156.0748 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7643	Non-flammable. Does not burn even when heated to its decomposition point. Does not form flammable mixtures with reducing agents, not even with aluminum powder.	Amazingly stable liquid. Non-sensitive to shock, percussion, heat, friction, and pressure; however, will detonate if rapidly heated under a high temperature. Requires blasting cap or detonator for initiation. A secondary explosive.	Generally non-reactive. Can be reduced with concentrated hydrochloric acid in the presence of finely divided tin. Non-reactive with chlorine, bromine, or iodine, but detonates when treated with fluorine. Stable to most oxidizers, but will detonate when mixed with potassium permanganate and	Colorless, syrupy liquid. Melting point: -45 Celsius; Boiling point: 210 Celsius under reduced pressure. Decomposes when heated to 308 Celsius; Insoluble in water, but moderately soluble in methylene chloride, hexane, and carbon disulfide.

			heated.	Relatively insoluble in ether, ethyl acetate, and alcohol.

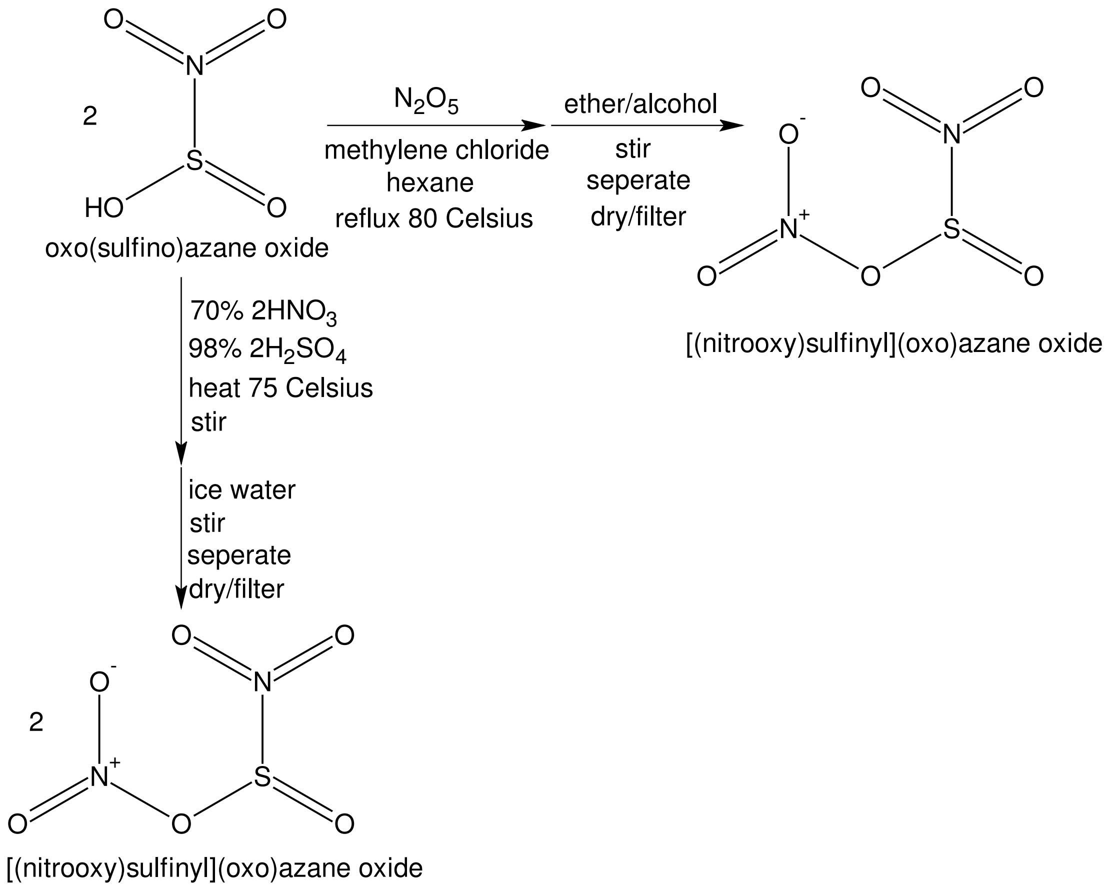
Estimated Compound summary: **Warning!** [(nitrooxy)sulfinyl](oxo)azane oxide produces severe migraines and headaches when vapors are inhaled, so use caution. [(nitrooxy)sulfinyl](oxo)azane oxide is a very stable liquid explosive with explosive properties closely related to nitroglycerine, without the notorious unstableness of nitroglycerine. [(nitrooxy)sulfinyl](oxo)azane oxide due to its stability can be used as an excellent substitute for nitroglycerine and other polyhydric nitroxy explosives. The only draw back is that it's a liquid and hence difficult to handle. It does not get absorbed by diatomaceous earth, but it can be absorbed by freshly precipitated aluminum hydroxide from which it forms a 60% mixture by weight (40% aluminum hydroxide). [(nitrooxy)sulfinyl](oxo)azane oxide will also form a slightly wet solid product when mixed with alcohol and sodium hydroxide added with stirring. Although [(nitrooxy)sulfinyl](oxo)azane oxide can be used alone as an explosive, its liquid status makes it difficult to deal with, so the two absorbed mixtures previously mentioned are only several ways to properly use the explosive. [(nitrooxy)sulfinyl](oxo)azane oxide can be slurried with finely divided TNT, picric acid, HMX, or PETN for making slurry explosives for blasting hard rock in above ground mining or road clearing operations. For military use, the two absorbed mixtures previously mentioned can be used in making blasting powders for demolition work. [(nitrooxy)sulfinyl](oxo)azane oxide, like nitroglycerine, can be used to make high performance double based smokeless powders as it is somewhat absorbed by nitrocellulose, but requires a small amount of sodium hydroxide to keep the [(nitrooxy)sulfinyl](oxo)azane oxide from leaching out of the gun propellant grains.

Safety Info: Extinguish all flames before using hexane, which is highly flammable. Use care when handling dinitrogen pentoxide, and avoid contact was combustible material. Use care when handling ether, which is highly volatile and forms explosive mixtures with air. Note: always perform the peroxide test before using ether that has been in storage for prolonged periods. Ether is a narcotic in high concentrations so use maximum ventilation when using. Wear gloves and avoid skin contact when handling concentrated nitric and sulfuric acids. Concentrated sulfuric acid can char many organic substances.

Materials:

1. 500 milliliters of dry hexane	7. 50 grams of anhydrous magnesium sulfate
2. 201 grams of oxo(sulfino)azane oxide (prepared in theoretical preparation 50, step 1)	8. or 160 grams of 70% nitric acid
3. 98 grams of dinitrogen pentoxide	9. or 200 grams of 98% sulfuric acid
4. 500 milliliters of methylene chloride	10. or 201 grams of oxo(sulfino)azane oxide (prepared in theoretical preparation 50, step 1)
5. 400 milliliters of diethyl ether	11. or 50 grams of anhydrous magnesium sulfate
6. 500 milliliters of 95% ethyl alcohol	

Reaction Equation:



Preparation:

Step 1: preparation 1 of [(nitrooxy)sulfinyl](oxo)azane oxide

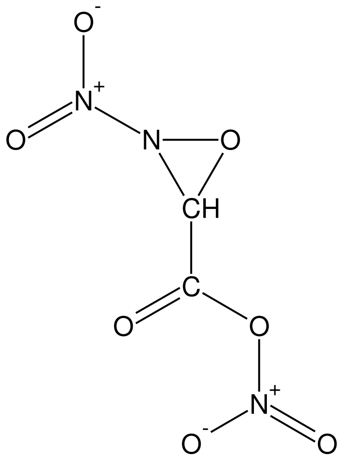
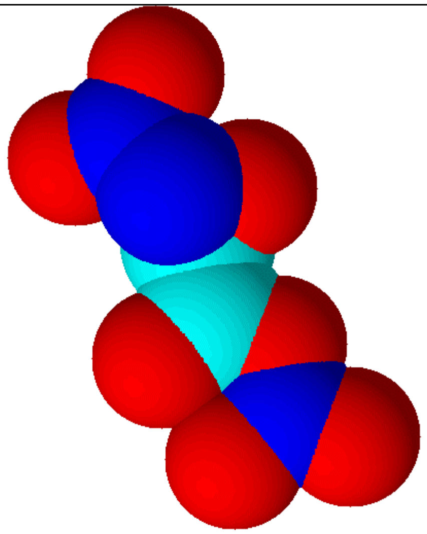
Into a standard reflux apparatus, using a three-neck flask sitting inside a round bottom heating mantle and equipped with motorized stirrer, reflux condenser, and addition funnel, place 500 milliliters of dry hexane, followed by 201 grams of oxo(sulfino)azane oxide (prepared in theoretical preparation 50, step 1). Thereafter, begin to stir the reaction mixture and at the same time, pour a methylene chloride solution of dinitrogen pentoxide prepared by dissolving 98 grams of dinitrogen pentoxide into 500 milliliters of methylene chloride into the addition funnel, and then turn on the heating mantle. When the temperature of the hexane/oxo(sulfino)azane oxide reaction mixture reaches 80 Celsius, under rapid stirring, begin to drip in the dinitrogen pentoxide methylene chloride solution into the reaction mixture containing the hexane/oxo(sulfino)azane oxide over a period of about 1 hour, while rapidly stirring the reaction mixture and maintaining its temperature around 80 Celsius. Once all the dinitrogen pentoxide/methylene chloride solution has been added to the reaction mixture, continue to reflux the reaction mixture at 80 Celsius under rapid stirring for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, turn off the motorized stirrer, and then disassemble the apparatus and pour the entire reaction mixture into a large beaker sitting on top of a magnetic stirrer, drop in a Teflon coated magnetic stir bar, and then turn on the magnetic stirrer device and begin to stir the reaction mixture. Immediately thereafter, pour in a solvent mixture prepared by adding and dissolving 400 milliliters of diethyl ether into 500 milliliters of 95% ethyl alcohol, and then continue to rapidly stir this new reaction mixture at room temperature for 1 hour. After 1 hour, turn off the magnetic stirrer, and pour the entire reaction mixture into a large separatory funnel and then allow it to stand for 1 hour, where upon a two phase mixture will result. After about 1 hour, carefully drain-off the bottom layer, which will be the desired product, and then pour the bottom layer into a clean beaker (this bottom layer will be the desired product of [(nitrooxy)sulfinyl](oxo)azane oxide) Note: if you do not have a super large separatory funnel simply use a smaller one and divide the reaction mixture into six or seven portions and repeat the separating process as many times as needed—combining each bottom product layer into a clean beaker each time. Now, once the bottom desired product layer has been collected into a clean beaker, throw in all at once 50 grams of anhydrous magnesium sulfate, and then manually stir this mixture for about 15 minutes to absorb any moisture that may be present.

Finally, after manually stirring for about 15 minutes, filter-off the magnesium sulfate, using suction filtration, and then collect the filtered liquid product of [(nitrooxy)sulfinyl](oxo)azane oxide, and store it in an amber glass bottle in a cool dry place until use.

Step 1: preparation 2 of [(nitrooxy)sulfinyl](oxo)azane oxide

Into a suitable sized beaker, sitting on top of a magnetic stirrer/hot plate, throw in a Teflon coated sir bar, and then add in 160 grams of 70% nitric acid, and thereafter, carefully and slowly pour in 200 grams of 98% sulfuric acid while rapidly stirring. Note: red fumes of toxic nitrogen oxides may evolve from this mixture, so use proper ventilation. Once all the concentrated sulfuric acid has been added to the nitric acid, continue to rapidly sir this nitrating acid reaction mixture, and then turn on the heating mantle, and heat this nitrating acid reaction mixture to 75 Celsius. Once the temperature reaches 75 Celsius, slowly add, at small quantities at a time, 201 grams of oxo(sulfino)azane oxide (prepared in theoretical preparation 50, step 1) over a period of about 1 hour, while rapidly stirring the reaction mixture and maintaining its temperature around 75 Celsius at all times. After the addition of the oxo(sulfino)azane oxide, continue to heat and stir the reaction mixture at 75 Celsius for 3 hours. After 3 hours, turn off the hot plate and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to stir the reaction mixture. Once the this reaction mixture has cooled to room temperature, stop stirring, and then slowly and carefully pour the entire reaction mixture into a large beaker filled with ice, and then allow the ice to melt—add more ice as needed during the addition. Note: use extreme caution when pouring concentrated sulfuric into water, as a great deal of heat is generated. Once all the reaction mixture has been added to ice, add more ice if needed, and then allow this new reaction mixture to stand for 6 hours. After 6 hours, a two-phase mixture will result. The desired product of [(nitrooxy)sulfinyl](oxo)azane oxide will be the upper layer. Now, pour the entire reaction mixture into a large separatory funnel and then allow it to stand for a few minutes to allow the two-phase mixture to form. Thereafter, carefully drain-off the bottom acid layer completely, and then pour the upper layer into a clean beaker (this upper layer will be the desired product of [(nitrooxy)sulfinyl](oxo)azane oxide) Note: if you do not have a super large separatory funnel simply use a smaller one and divide the reaction mixture into six or seven portions and repeat the separating process as many times as needed—combining each upper product layer into a clean beaker each time. Now, once the upper desired product layer has been collected, throw in all at once 50 grams of anhydrous magnesium sulfate, and then manually stir this mixture for about 15 minutes to absorb any moisture that may be present. Finally, after manually stirring for about 15 minutes, filter-off the magnesium sulfate, using suction filtration, and then collect the filtered liquid product of [(nitrooxy)sulfinyl](oxo)azane oxide, and store it in an amber glass bottle in a cool dry place until use.

Theoretical Preparation 52: (2-nitrooxaziridin-3-yl)carbonyl nitrate; 2-NOCN; Nitroaziridin nitrate carbonyl; KAT-12

 (2-nitrooxaziridin-3-yl)carbonyl nitrate	
Expanded structure	3D structure

Molecular data:
Molecular Formula = $C_2HN_3O_7$
Formula Weight = 179.04524
Composition = C(13.42%) H(0.56%) N(23.47%) O(62.55%)

Molar Refractivity = 28.79 ± 0.4 cm^3
Molar Volume = 85.4 ± 5.0 cm^3
Parachor = 268.2 ± 6.0 cm^3
Index of Refraction = 1.588 ± 0.03
Surface Tension = 97.1 ± 5.0 dyne/cm
Density = 2.09 ± 0.1 g/cm^3
Dielectric Constant = Not available
Polarizability = 11.41 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 178.981449 Da
Nominal Mass = 179 Da
Average Mass = 179.0452 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6895	Flammable solid; burns with a non-violent flame emitting toxic fumes of nitrogen oxides and carbon monoxide. Mixtures with reducing agents ignite rapidly and burn violently i.e see potassium nitrate.	Very stable. Non-sensitive to shock, percussion, heat, pressure and friction. A secondary explosive. Requires a blasting cap or detonator for initiation.	Mostly stable to chemical attack. Is not attacked by halogens except for fluorine, which causes the compound to ignite (self deflagrate). Is not attacked by strong oxidizers even potassium permanganate.	Light lemon yellowish crystalline powder. Melting point: 198 Celsius; Boiling point: Decomposes when heated to 345 Celsius. Insoluble in water and most organic solvents. Has limited solubility in carbon disulfide and liquid ammonia. Slightly soluble in concentrated sulfuric acid

Estimated Compound summary: (2-nitrooxaziridin-3-yl)carbonyl nitrate is an interesting oxaziridine nitroamine-nitrate explosive with remarkable stability. As with many other stable secondary explosives, it can be melted down and poured into molds for making shape charges, linear shape charges, and poured into mortar shells, artillery shells, and/or poured into molds for making a variety of warheads for rockets and missiles. Unlike other oxaziridine explosives, (2-nitrooxaziridin-3-yl)carbonyl nitrate when molten is soluble in a variety of other high explosives such as nitro benzenes, nitro triazoles and tetrazoles, and nitro phenyls; however, (2-nitrooxaziridin-3-yl)carbonyl nitrate when molten is insoluble in cyclic nitramine explosives and polyhydric explosives such as RDX, HMX, PETN, ect., (2-nitrooxaziridin-3-yl)carbonyl nitrate when melted and mixed with powdered aluminum and then cured with epoxy, a powerful high performance rocket fuel is created that can be used to propel missiles and rockets for various military and commercial operations. When (2-nitrooxaziridin-3-yl)carbonyl nitrate is slurried in nitroglycerine or any other polyhydric liquid explosive, and then treated with a high energy plasticizer, a moldable, foldable, matt like explosive composition results, that can be used for demolition operations, blasting operations, or for destroying old ageing military ordnances. (2-nitrooxaziridin-3-yl)carbonyl nitrate can also be used in making high performance gun propellants when slurried with nitroglycerine or any other polyhydric liquid explosive and then mixed with nitrocellulose and potassium nitrate. As mentioned in molecular information, when (2-nitrooxaziridin-3-yl)carbonyl nitrate is mixed in correct proportions with reducing agents, i.e., combustible materials, mixtures result that ignite readily by a flame or spark and burn violently, hence, these mixtures can be used to make pyrotechnic compositions for a variety of uses. (2-nitrooxaziridin-3-yl)carbonyl nitrate should not be detonated in large quantities in enclosed areas due to toxic fumes produced upon detonation.

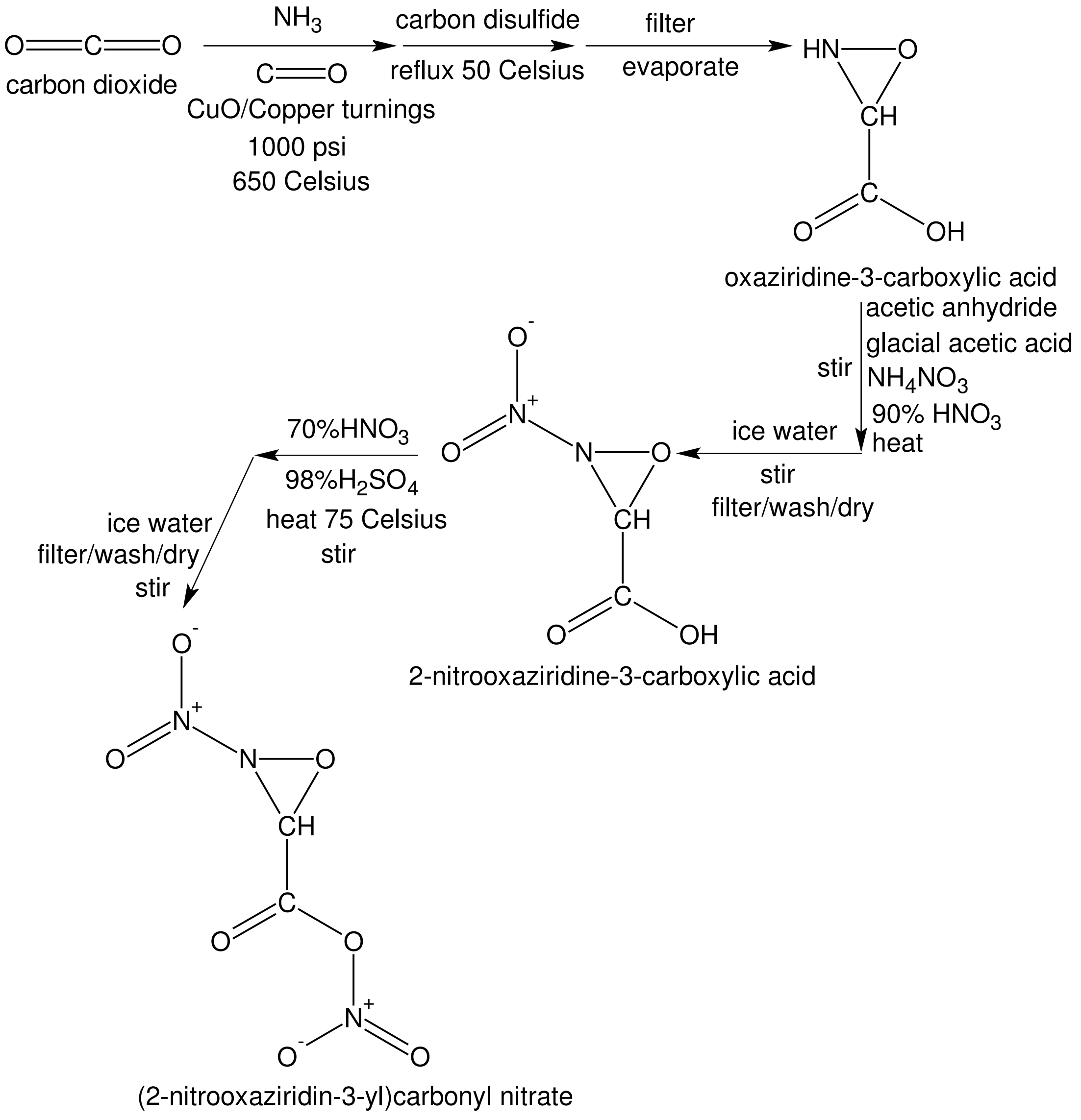
Safety Info: Use proper ventilation and avoid inhalation, and eye contact of ammonia gas, which is highly irritating to eyes, nose, and throat. Use proper care and maximum ventilation when handling carbon monoxide, which is highly toxic. Avoid inhalation, ingestion, and skin contact when handling carbon disulfide, which is readily absorbed by the body and is very toxic.

Wear gloves when handling glacial acetic acid and acetic anhydride, as they are corrosive and irritating. Use maximum caution and ventilation when handling 90% nitric acid, which evolves toxic fumes of nitrogen oxides. Wear gloves and avoid skin and clothing contact when handling 70% nitric and an especially 98% sulfuric acid, the latter is highly corrosive and can char many organic substances.

Materials:

1. 190 grams of copper-II-oxide	8. 500 milliliters of glacial acetic acid
2. 300 grams of copper turnings	9. 600 milliliters of acetic anhydride
3. Argon gas from tank	10. 89 grams of ammonium nitrate
4. 127 grams of carbon dioxide gas	11. 200 grams of a 90% nitric acid
5. 49 grams of dry ammonia gas	12. 1,411 grams of 70% nitric acid
6. 81 grams of carbon monoxide gas	13. 2000 milliliters of 98% sulfuric acid
7. 750 milliliters of triple distilled carbon disulfide	14. 3000 milliliters of a 10% sodium carbonate solution

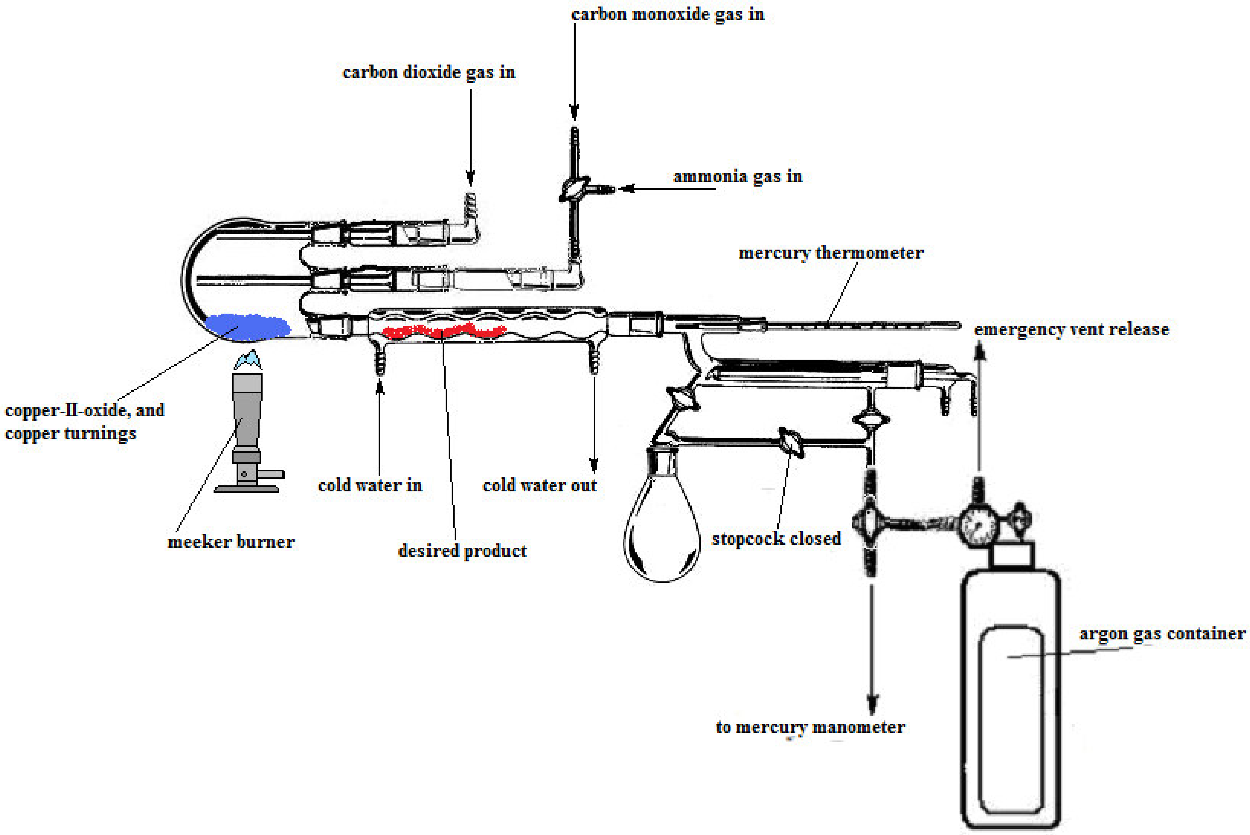
Reaction Equation:



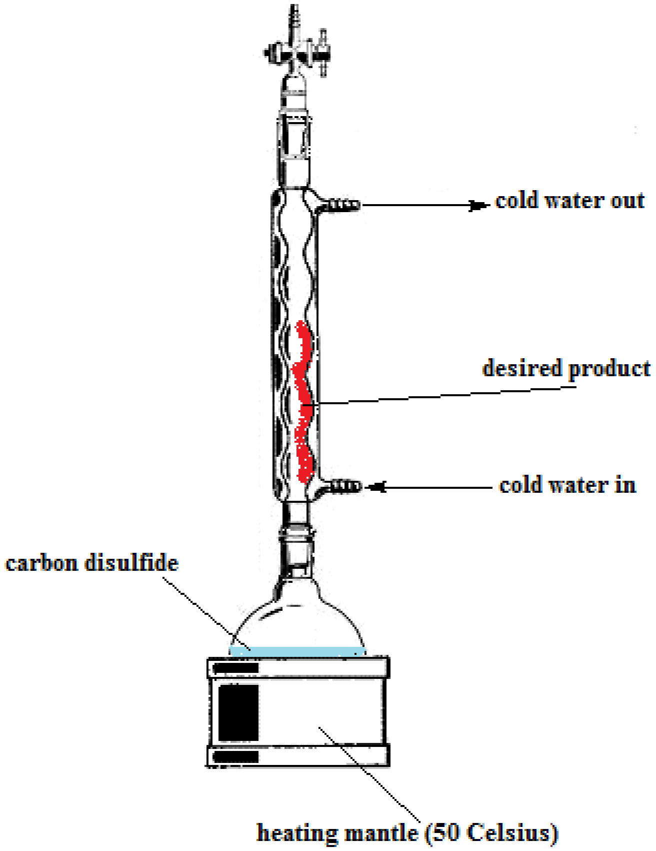
Preparation:

Step 1: preparation of oxaziridine-3-carboxylic acid

Into an apparatus as illustrated below, place 190 grams of copper-II-oxide and then place 300 grams of copper turnings, and then begin a argon gas purge, and at the same time, turn on the meeker burner, and heat the copper-II-oxide and copper turnings to 650 Celsius. Thereafter, bring the pressure inside the apparatus to 1000 psi. Once the copper-II-oxide/copper turnings have reached 650 Celsius, and the apparatus has a pressure of 1000 psi, begin to pass into the reaction chamber carbon dioxide gas at a flow rate of 50 mg per/minute. At the same time begin to pass into the reaction chamber dry ammonia gas at a flow rate of 20 mg per/minute, and begin to pass in dry carbon monoxide at a flow rate of 32 mg per/minute while heating the reaction chamber to 650 Celsius while maintaining a pressure inside the apparatus at 1000 psi. Continue to heat the reaction chamber as illustrated to 650 Celsius, while maintaining a pressure inside the reaction apparatus at 1000 psi until approximately 127 grams of carbon dioxide gas has been added, and 49 grams of dry ammonia gas has been added, and 81 grams of carbon monoxide gas has been passed into the reaction chamber. Once all the gram quantities of all three gasses has been passed into the reaction chamber, turn off the meeker burner and release the pressure gauge to vent the argon gas purge and bring the inside of the apparatus back to standard atmospheric pressure. Once the reaction chamber has cooled to room temperature, disassemble the apparatus, and then take the bulb condenser containing the desired product stuck to the walls thereof, and then assemble a reflux apparatus as illustrated in the second illustration. Thereafter, pour in 750 milliliters of triple distilled carbon disulfide, and then turn on the heating mantle and begin to reflux the carbon disulfide at 50 Celsius. During the reflux process, the carbon disulfide will dislodge and dissolve the desired product of oxaziridine-3-carboxylic acid. Continue to reflux this carbon disulfide for 4 hours, until all the product stuck to the walls of the bulb condenser have been removed. Once this point is achieved, turn off the heating mantle, and allow the carbon disulfide mixture to cool to room temperature. Finely, once the carbon disulfide mixture has cooled to room temperature, filter the mixture to remove insoluble materials using suction filtration, and then take the filtered carbon disulfide mixture and place it into a rotary evaporator, and evaporate-off the carbon disulfide under moderate vacuum until dry solid remains. Note: carbon disulfide is toxic so use maximum and proper ventilation. When dry solid remains, turn off the vacuum and the rotary evaporator, and recover this left over dried residue of oxaziridine-3-carboxylic acid, and save for step 2.



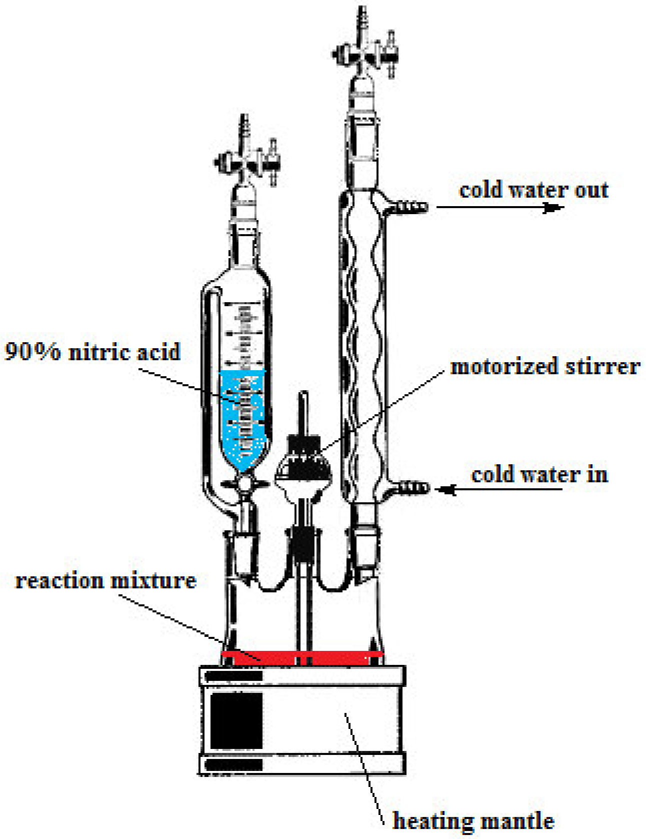
All nickel apparatus for preparing oxaziridine-3-carboxylic acid.



Setup for refluxing carbon disulfide to dislodge and dissolve the desired product of oxaziridine-3-carboxylic acid.

Step 2: preparation of 2-nitrooxaziridine-3-carboxylic acid

Into an apparatus as similar to one illustrated below, place the dried product of oxaziridine-3-carboxylic acid (prepared in step 1), and then add in 500 milliliters of glacial acetic acid, followed by 600 milliliters of acetic anhydride, and then followed by 89 grams of ammonium nitrate. Immediately thereafter, pour into the addition funnel 200 grams of a 90% nitric acid solution. Now, turn on the heating mantle, and motorized stirrer and begin to rapidly stir the reaction and turn the heating mantle to 50 to 60 Celsius. When the reaction mixtures temperature reaches 50 to 60 Celsius, under rapid stirring, begin to slowly drip into the reaction mixture the 90% nitric acid from the addition funnel. Continue to heat the reaction mixture to 50 to 60 Celsius under rapid agitation until all the 90% nitric acid has been added to the reaction mixture. Once all the 90% nitric acid has been added, continue to heat the reaction mixture at 50 to 60 Celsius under rapid agitation for 3 hours. After 3 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, vigorously stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour the entire reaction mixture into a large clean beaker filled with ice water, and then stir this new mixture for about 2 hours. After 2 hours, filter-off the insoluble desired product of 2-nitrooxaziridine-3-carboxylic acid using suction filtration, and then wash with six 500-milliliter portions of warm water, and then suction dry as best as possible. Thereafter, take the filtered-off washed desired product of 2-nitrooxaziridine-3-carboxylic acid and store it in a dessicator over sodium hydride for 7 days. After 7 days, the desired product of 2-nitrooxaziridine-3-carboxylic acid is ready for step 3.

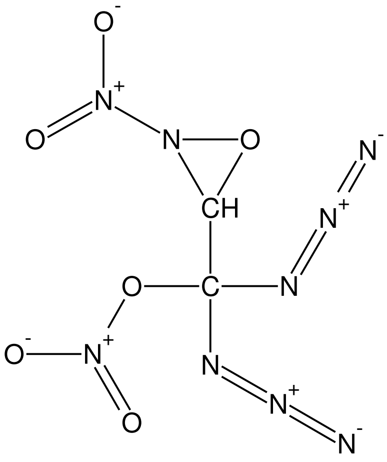
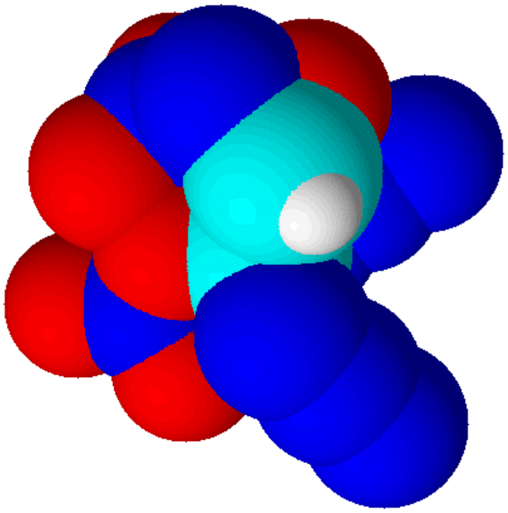


Setup for first nitration of oxaziridine-3-carboxylic acid.

Step 3: preparation of (2-nitrooxaziridin-3-yl)carbonyl nitrate

Into a standard reflux apparatus with large 3-neck flask sitting inside a heating mantle, and equipped with motorized stirrer, reflux condenser and thermometer, place the dried product of 2-nitrooxaziridine-3-carboxylic acid (obtained in step 2), and then add in 1,411 grams of 70% nitric acid, and then carefully and slowly add in 2000 milliliters of 98% sulfuric acid. Then begin to rapidly stir this reaction mixture, and then turn on the heating mantle and heat the reaction mixture to 75 Celsius. When the reaction mixtures temperature reaches 75 Celsius, continue to stir the reaction mixture for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during the cool down period continue to vigorously stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then pour entire reaction mixture slowly, and carefully pour this entire reaction mixture into a super large beaker filled with ice water, and stir this mixture for about 6 hours, adding more ice as needed during this 6 hour period. After 6 hours, filter-off the insoluble desired product of (2-nitrooxaziridin-3-yl)carbonyl nitrate using suction filtration, and then wash first, with six 500-milliliter portions of a 10% sodium carbonate solution, and then wash with six 750-milliliter portions of hot water, and then suction dry as best as possible. Finely, place the suction dried filtered-off desired product of (2-nitrooxaziridin-3-yl)carbonyl nitrate into an oven, and heat to 110 Celsius for 12 hours. After 12 hours, turn off the oven, and allow the dried desired product to cool to room temperature. Once it has, remove it from the oven, and store in a plastic container unless use.

Theoretical Preparation 53: diazido(2-nitrooxaziridin-3-yl)methyl nitrate; Nitrooxaziridin diazide nitrate; 2-DNAMN; KL-55

 O N O N O CH N N N O C N O N O N N N diazido(2-nitrooxaziridin-3-yl)methyl nitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_2HN_9O_6$
Formula Weight = 247.08604
Composition = C(9.72%) H(0.41%) N(51.02%) O(38.85%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 247.004979 Da
Nominal Mass = 247 Da
Average Mass = 247.086 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7890; estimated using infrared spectrometry.	Non-flammable; Does not burn even when heated. Begins to fizzle when heated to 350 Celsius evolving suffocating fumes. Does not form flammable mixtures even when mixed with reducing agents.	Very stable. Non-sensitive to shock, percussion, heat, pressure, or fiction. A secondary explosive. Requires a blasting cap or detonator for initiation.	Virtually non-reactive. Does not react with halogens except fluorine, in which it inflames. Has great resistance to strong oxidizers, and does not react with potassium permanganate even when heated.	Colorless, crystalline solid, or white powder. Melting point: 201 Celsius, Boiling point: decomposes when heated to 350 Celsius. Insoluble in water, and all known solvents.

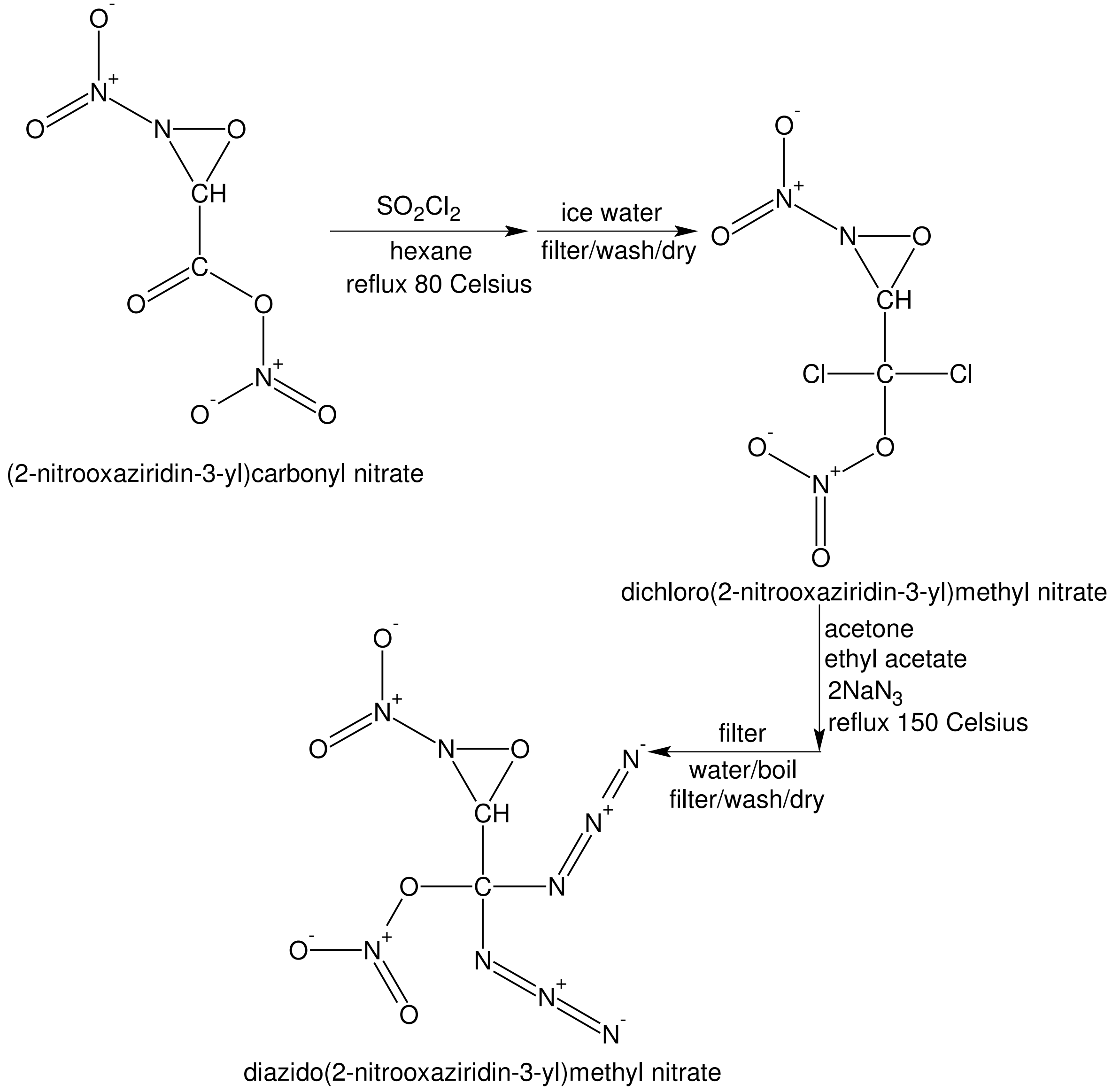
Compound summary: diazido(2-nitrooxaziridin-3-yl)methyl nitrate is an interesting and peculiar nitro, nitrate, and azide containing high explosive with a relatively and surprisingly high melting point, but moderate decomposition point. Like many experimental explosives, it is insoluble when molten, in almost all know molten high explosives, but it can be poured, when molten into molds for making shape charges or for filling mortar shells, artillery shells, bomb casings, or for molding into any desired body for making demolition charges, missile warheads, land mines, and any other desired explosive body for many different types of military and commercial operations. diazido(2-nitrooxaziridin-3-yl)methyl nitrate either when first molten and then casted, or when in powdered form, requires a significant blasting cap or detonator preferably of the RDX type for proper initiation—this is caused by the stereo bonds between the azide groups and the chiral carbon making diazido(2-nitrooxaziridin-3-yl)methyl nitrate very stable towards shock even molecular shock. In any sense, diazido(2-nitrooxaziridin-3-yl)methyl nitrate is an interesting high explosive that warrants further investigation; however, other then use as a high explosive, it has little potential for use in making pyrotechnic compositions, as it demonstrates little action when mixed with combustible materials, even when mixed with powdered aluminum and ignited. Nonetheless, diazido(2-nitrooxaziridin-3-yl)methyl nitrate can be used to form high performance gun propellants when it is mixed with liquid explosives of the polyhydric type, i.e., nitroglycerine and then treated with a high energy plasticizer, and then cured. These high energy gun propellants should only be used in thick bored guns such as deck guns, or high caliber guns as the pressures generated upon ignition of the said propellants can exceed 100,000 psi, causing small arms weapons to blow up when fired. But when these high-energy gun propellants are used under the appropriate conditions, they have tremendous heaving power and can heave heavy shells many kilometers.

Safety Info: Extinguish all sources if ignition when handling hexane, which is highly flammable. Wear gloves, and avoid skin contact and contact with moisture when handling sulfuryl chloride, which can cause severe skin irritation and reacts vigorously with water. Use proper ventilation when handling acetone and ethyl acetate, and avoid inhalation of the fumes, and keep in mind both solvents are highly flammable. Wear gloves and avoid skin contact and ingestion when handling sodium azide, which is highly toxic. Use maximum ventilation when handling ether, which forms explosive mixtures with air. Avoid inhalation of ether fumes, as ether is a narcotic in high concentrations. Also, never use ether that has been in prolonged storage unless you perform the peroxide test first.

Materials:

1. 137 grams of (2-nitrooxaziridin-3-yl)carbonyl nitrate (obtained in theoretical preparation 52, step 3),	5. 500 milliliters of acetone
2. 500 milliliters of dry hexane	6. 550 milliliters of ethyl acetate
3. 103 grams of sulfuryl chloride	7. 96 grams of sodium azide
4. 1500 milliliters of a 10% sodium carbonate solution	8. 3000 milliliters of fresh diethyl ether

Reaction Equation:



SO2Cl2
hexane
reflux 80 Celsius
ice water
filter/wash/dry
(2-nitrooxaziridin-3-yl)carbonyl nitrate
dichloro(2-nitrooxaziridin-3-yl)methyl nitrate
acetone
ethyl acetate
2NaN3
reflux 150 Celsius
filter
water/boil
filter/wash/dry
diazido(2-nitrooxaziridin-3-yl)methyl nitrate

Preparation:

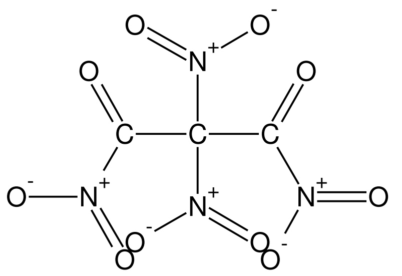
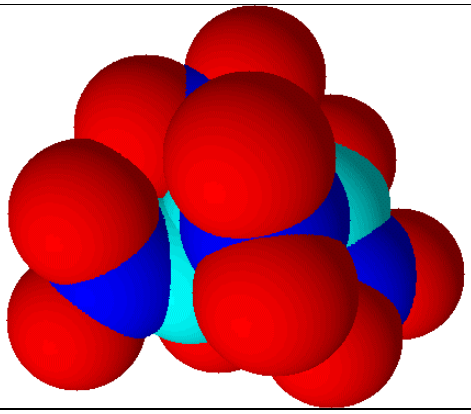
Step 1: preparation of dichloro(2-nitrooxaziridin-3-yl)methyl nitrate

Into a standard dry reflux apparatus utilizing a suitable sized 3-neck flask fitted inside an appropriate heating mantle, and this 3-neck flask equipped with addition funnel, motorized stirrer, reflux condenser, and thermometer, place 137 grams of (2-nitrooxaziridin-3-yl)carbonyl nitrate (obtained in theoretical preparation 52, step 3), and then pour in 500 milliliters of dry hexane. Thereafter, turn on the heating mantle, and bring the contents in the 3-neck flask to a gentle reflux at 80 Celsius under vigorous stirring. When the temperature of the contents in the 3-neck flask reach 80 Celsius under vigorous stirring, pour into the addition funnel, 103 grams of sulfuryl chloride, and then begin a slow drip of this sulfuryl chloride from the addition funnel into the 3-neck flask while maintaining a reflux of 80 Celsius under vigorous stirring. Continue to slowly drip into the 3-neck flask the sulfuryl chloride from the addition funnel while maintaining a reflux of 80 Celsius under vigorous stirring until all the sulfuryl chloride has been added. Once all the sulfuryl chloride has been added to the reaction mixture, close the stopcock on the addition funnel, and then continue to gently reflux the reaction mixture at 80 Celsius under vigorous stirring for 5 hours. After 5 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: continue to vigorously stir the reaction mixture during this cool down period. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then slowly and carefully pour the entire reaction mixture into 1500 milliliters of ice water contained in a clean large beaker equipped with a motorized stirrer (not a magnetic stirrer), and once all the reaction mixture has been poured into the ice water, turn on the motorized stirrer and rapidly blend this new mixture on high speed for 2 hours. After 2 hours, turn off the motorized stirrer, and then filter-off the insoluble desired product of dichloro(2-nitrooxaziridin-3-yl)methyl nitrate using suction filtration, and then wash this filtered-off desired product with three 500-milliliter portions of a 10% sodium carbonate solution, followed by washing with three 500-milliliter portions of hot water, and then suction dry as best as possible, and then store the washed suction dried desired product of dichloro(2-nitrooxaziridin-3-yl)methyl nitrate in a dessicator over concentrated sulfuric acid for 7 days before using in step 2.

Step 2: preparation of diazido(2-nitrooxaziridin-3-yl)methyl nitrate

Into a clean reflux apparatus utilizing an appropriate sized 3-neck flask sitting inside a heating mantle, and this 3-neck flask equipped with reflux condenser, motorized stirrer, and thermometer, place into this 3-neck flask the product of dichloro(2-nitrooxaziridin-3-yl)methyl nitrate obtained in step 1, followed by 500 milliliters of acetone, followed by 550 milliliters of ethyl acetate, and then followed by 96 grams of sodium azide. Thereafter, turn on the motorized stirrer, and then turn on the heating mantle to 150 Celsius, and begin a vigorous reflux under vigorous stirring for 6 hours. After 6 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to vigorously stir the reaction mixture. Once the reaction mixture has cooled to room temperature, turn off the motorized stirrer, and then disassemble the apparatus, and then filter-off the insoluble material using suction filtration, and then suction dry as best as possible. Now, take this suction dried filtered-off material, which will contain the by-product sodium chloride, and then place it into a clean beaker sitting on top of a hot plate, and then add in 2000 milliliters of ordinary tap water. Thereafter, turn on the hot plate to 100 Celsius, and bring the mixture to a boil. Once the mixture begins to boil, continue to boil the mixture to dissolve the by-product sodium chloride for 1 hour. After 1 hour, turn off the hot plate, and allow the mixture to cool to about 50 Celsius. When its temperature reaches 50 Celsius, filter-off (using suction filtration) the remaining insoluble material, which will be the desired product of diazido(2-nitrooxaziridin-3-yl)methyl nitrate, and then wash with six 500-milliliter portions of fresh diethyl ether, followed by washing with six 500-milliliter portions of hot water, and then suction dry as best as possible. Finely, take this filtered-off washed desired product, and place it into an oven, and bake at 105 Celsius for 12 hours. After 12 hours, turn off the oven, and allow the dried desired product to cool to room temperature. Once it has, remove it from the oven, and store in a plastic container in a cool dry place until use.

Theoretical Preparation 54: 1,2,2,3-tetranitro-1,3-dioxopropane; Nitro dioxopropane; Tetranitro dioxopropane; TNDOP; 1,2,2,3-TNDOP;

 1,2,2,3-tetranitro-1,3-dioxopropane	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_3N_4O_{10}$
Formula Weight = 252.0529
Composition = C(14.30%) N(22.23%) O(63.48%)
Molar Refractivity = 40.06 ± 0.3 cm^3
Molar Volume = 123.4 ± 3.0 cm^3
Parachor = 390.9 ± 4.0 cm^3
Index of Refraction = 1.562 ± 0.02
Surface Tension = 100.5 ± 3.0 dyne/cm
Density = 2.041 ± 0.06 g/cm^3
Dielectric Constant = Not available
Polarizability = 15.88 ± 0.5 $10^{-24}cm^3$

Monoisotopic Mass = 251.961442 Da
Nominal Mass = 252 Da
Average Mass = 252.0529 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7699	Non-flammable at ordinary temperatures, but will burn very slowly emitting highly irritating fumes burning with a sooty flame when heated to 120 Celsius and ignited using a flame. Will not ignite if touched by a spark or electric arc. Will only form flammable mixtures with powdered metals mainly powdered aluminum or powdered magnesium, but even then, these mixtures do not burn completely and burn emitting soot.	Very stable. Non-sensitive to shock, percussion, heat, pressure, and friction. A secondary explosive. Requires a blasting cap or detonator for initiation.	Virtually non reactive. Does not react with strong bases, or strong oxidizers even when heated. Does not react with halogens except for fluorine as usual with ignition of the compound burning with a sooty flame with sporadic explosions.	Yellowish syrupy liquid or light yellowish-red solid. Melting point 8.7 Celsius, Boiling point: 198 Celsius; decomposes when heated to 546 Celsius forming nitrogen oxides and carbon soot. Insoluble in water but has amazing solubility in toluene, methylene chloride, hexane, with slight solubility in acetone, and carbon disulfide; Insoluble in ether, liquid ammonia, and THF. **Warning!** 1,2,2,3-tetranitro-1,3-dioxopropane is highly irritating to the eyes, nose, and throat; use caution.

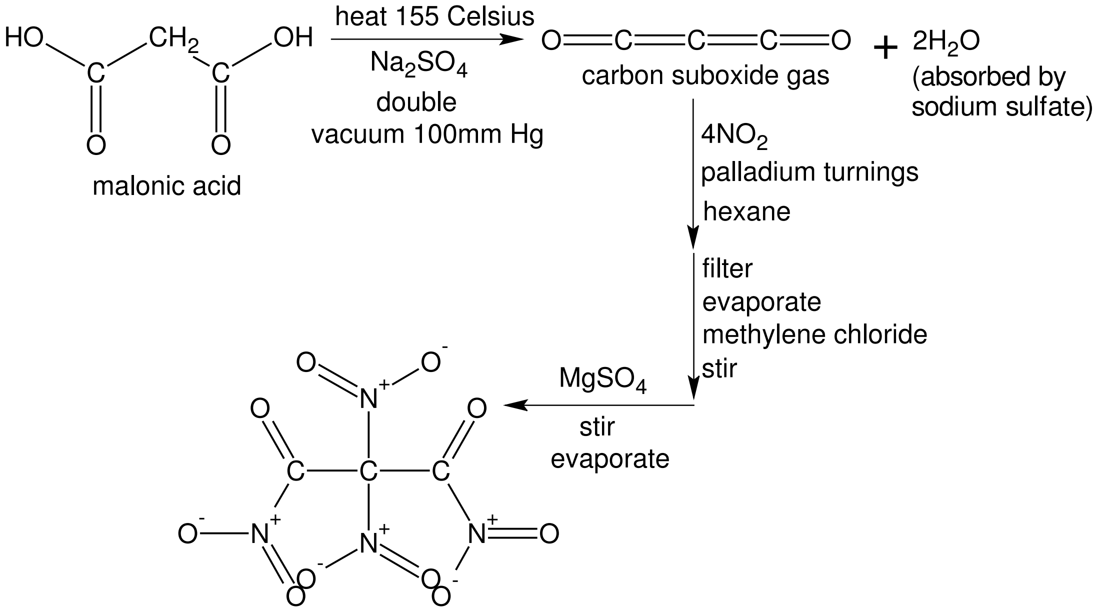
Estimated Compound summary: 1,2,2,3-tetranitro-1,3-dioxopropane is a highly irritating syrupy liquid explosive at room temperature, which should be handled with care as it is highly irritating to the nose, throat, and eyes. It also produces severe long lasting migraines when its fumes or vapors are inhaled, so caution should be taken and proper safety precautions utilizing maximum ventilation and proper storage should be exercised when dealing with this explosive. 1,2,2,3-tetranitro-1,3-dioxopropane is an interesting syrupy liquid high explosive that is not suitable to be used by-itself as an explosive (even though it can be when properly primed), but it forms a unique solid explosive when slurried with nitrocellulose and a fluorine containing high energy plasticizer. This gummy-plumbers putty like solid mass makes it very useful as a plastic explosive. This plastic explosive can be used in almost any desired means; it can be pressed into mortar shells, artillery shells, claymores, land mines, torpedoes, missile warheads, rocket warheads, demolition blocks, and shape charges for use in making a variety of munitions for multiple military operations. When 1,2,2,3-tetranitro-1,3-dioxopropane is mixed with nitrocellulose, mixed with powdered aluminum, and then mixed with a double base oxidizer such as ammonium perchlorate and potassium nitrate, it forms a unique dough like material, that can be extruded into any desired grains for use as a high performance gun propellant for smalls arms and even high caliber guns such as battleship deck guns. In an off note, 1,2,2,3-tetranitro-1,3-dioxopropane can be used a chemical warfare agent when it is mixed with a liquefied inert petroleum gas and contained in a compressed container with a bursting charge. 1,2,2,3-tetranitro-1,3-dioxopropane has absolutely no use for making pyrotechnic mixtures, or for making rocket fuels.

Safety Info: Extinguish all sources of ignition when handling hexane, which is highly flammable. Use proper care when handling nitrogen dioxide gas, which is highly toxic, and corrosive.

Materials:

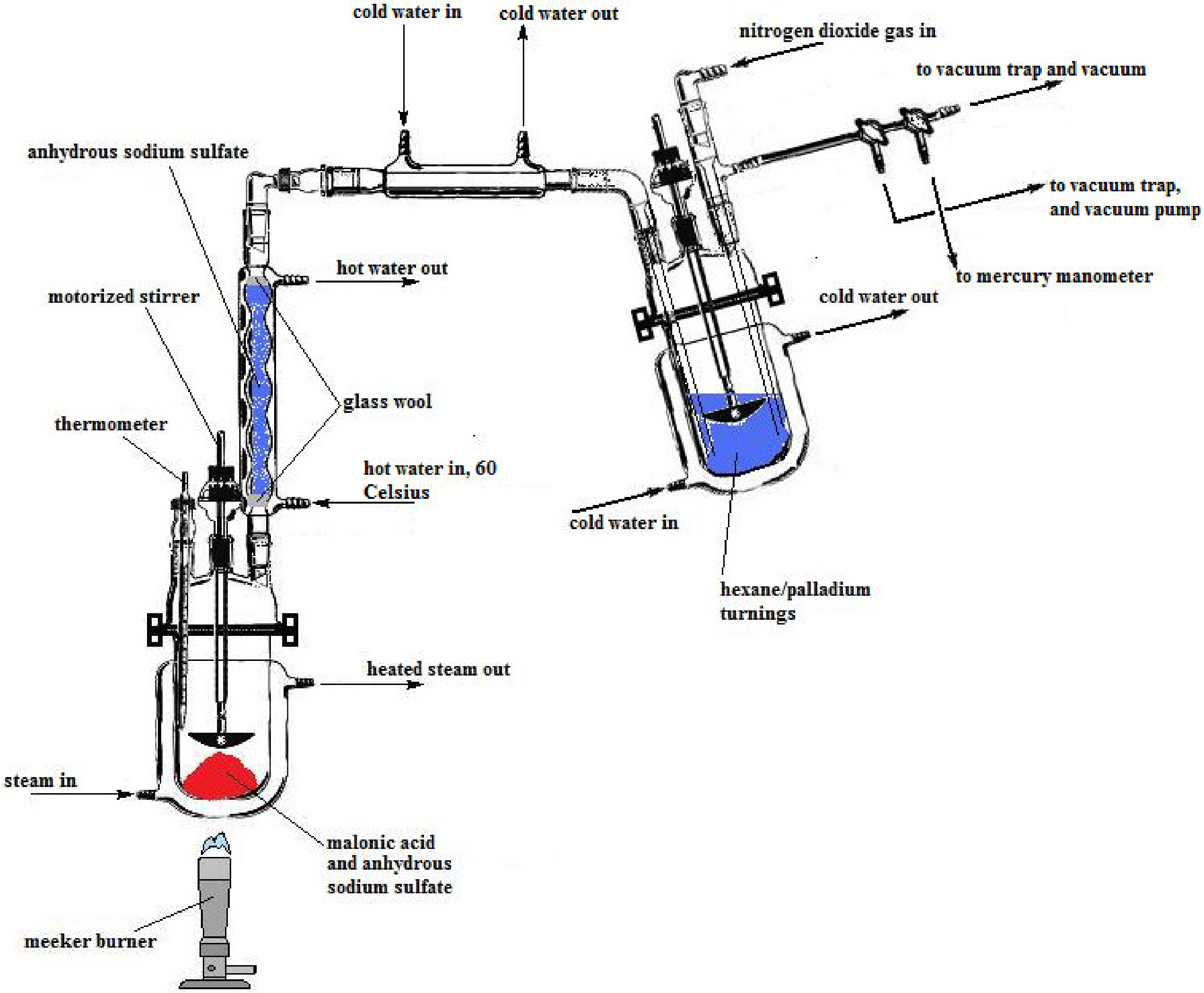
1. 209 grams of malonic acid	4. 395 grams of dry nitrogen dioxide gas
2. 600 grams of anhydrous magnesium sulfate	5. 700 milliliters of methylene chloride
3. 750 milliliters of dry hexane	

Reaction Equation:



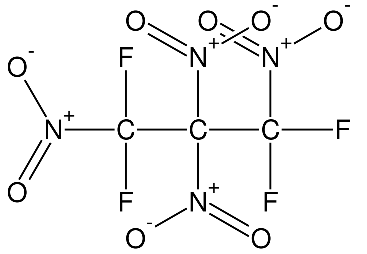
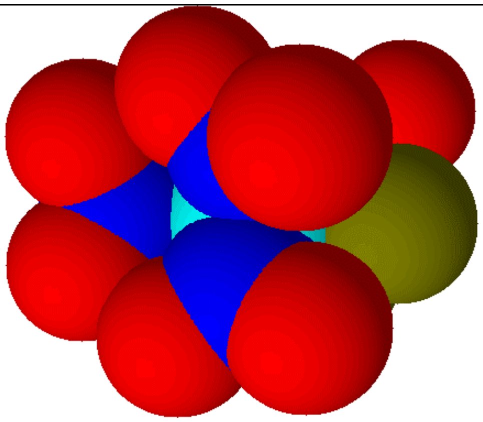
1,2,2,3-tetranitro-1,3-dioxopropane

Preparation: Into a radical and special apparatus as illustrated below, place 209 grams of malonic acid, followed by 250 grams of anhydrous magnesium sulfate into the reaction container as illustrated, and then turn on the motorized stirrer as illustrated. Then, into the second reaction container shown to the right of the illustration, pour in 750 milliliters of dry hexane, and then turn on this motorized stirrer as shown in the illustration, and then begin to pass in cold water through the outer jacket of this second reaction container as illustrated. Thereafter, place 300 grams of anhydrous magnesium sulfate granules into the bulb condenser as illustrated. Now, using a separate means for producing steam (not illustrated) begin to pass in steam through the outer jacket of the first reaction container as illustrated, and then turn on the meeker burner, and raise the temperature of the malonic acid/magnesium sulfate mixture to about 155 Celsius. Immediately thereafter, turn on the two vacuum pumps as declared in the illustration, and reduce the pressure inside the apparatus to 100 millimeters of mercury. Once the pressure inside the apparatus reaches 100 millimeters of mercury, and when the contents of the malonic acid/magnesium sulfate in the reaction container reaches 155 Celsius, a chemical reaction will begin, and a compound called carbon suboxide will begin to steadily form from the break down of the malonic acid……….now wait 5 minutes at this point, and then begin to pass into the second reaction container to the right as illustrated, dry nitrogen dioxide gas at a flow rate of 2.7 grams per/minute while rapidly stirring the contents in the second reaction container as illustrated. Now, continue to heat the malonic acid/magnesium sulfate reaction mixture in the first reaction container as illustrated until no more gas is seen emanating from the reaction mass. Also, during the break down of the malonic acid to produce carbon suboxide, continue to bubble into the hexane in the second reaction container with stirring, nitrogen dioxide gas until 395 grams of dry nitrogen dioxide gas has been added. Note: once all the malonic acid has broken down forming carbon suboxide, most of the dry nitrogen dioxide gas should have been added, and hence reacted with the carbon suboxide to produce the desired product, which will dissolve in the hexane. Once no more gas is seen emanating from the reaction mass in the first reaction container as illustrated, turn off the steam source, and turn off the meeker burner, and then finish bubbling in the remaining nitrogen dioxide gas into the second reaction container as illustrated with stirring and cooling until all the nitrogen dioxide gas has been added. Once all the nitrogen dioxide gas has been added, turn off both vacuum pumps, and disconnect them from the apparatus and allow the pressure inside the reaction apparatus to reach standard atmospheric pressure. Now, while continuing to pump cold water into the outer jacket of the second reaction container, continue to stir this hexane reaction mixture for 1 hour at STP. Now, after 1 hour, turn off the cold water cooling system, and then disassemble the apparatus, and then quickly filter the hexane reaction mixture from the second reaction container (to remove any insoluble impurities; mostly polymerized impurities) using suction filtration, and then take this filtered hexane reaction mixture and place it into a large rotary evaporator, and evaporate-off the hexane at 50 Celsius under moderate vacuum until no more hexane passes over, and a syrupy liquid remains. When no more hexane passes over, turn off the rotary evaporator, and then recover the left over syrupy liquid product left behind and place this syrupy liquid into a clean beaker (using proper ventilation), and then pour in 700 milliliters of methylene chloride, and then stir this mixture to dissolve the recovered syrupy liquid product. Continue to stir this mixture for 1 hour at room temperature using a magnetic stirrer or motorized stirrer, and then after 1 hour, add in 50 grams of anhydrous magnesium sulfate, and then continue to stir this new mixture for another 1 hour. After this additional 1-hour period, stop the stirring, and then filter this new mixture to remove the insoluble magnesium sulfate and other impurities using suction filtration. Finally, pour this filtered methylene chloride mixture into a clean rotary evaporator, and evaporate off the methylene chloride at 40 Celsius under mild vacuum until no more methylene chloride passes over. Once no more methylene chloride passes over, turn off the rotary evaporator, and recover the syrupy liquid product left behind, and then place this recovered syrupy liquid product of 1,2,2,3-tetranitro-1,3-dioxopropane into an amber glass bottle and store in a refrigerator under proper ventilation until use.



Advanced apparatus for the nitration of carbon suboxide.

Theoretical Preparation 55: 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane; TFTNP; Polyfluoro polynitropropane; Fluoro nitro propane-107; tetra-tetra; Cl-901

Expanded structure	3D structure
 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane	

Molecular data:

Molecular Formula = $C_3F_4N_4O_8$
Formula Weight = 296.0477128
Composition = C(12.17%) F(25.67%) N(18.92%) O(43.24%)
Molar Refractivity = 40.59 ± 0.3 cm^3
Molar Volume = 151.1 ± 3.0 cm^3
Parachor = 409.0 ± 4.0 cm^3
Index of Refraction = 1.449 ± 0.02
Surface Tension = 53.6 ± 3.0 dyne/cm
Density = 1.959 ± 0.06 g/cm^3
Dielectric Constant = Not available
Polarizability = 16.09 ± 0.5 $10^{-24}cm^3$
Monoisotopic Mass = 295.965226 Da
Nominal Mass = 296 Da
Average Mass = 296.0477 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7905; estimated using HPGC	Flammable; burns violently in the presence of oxygen. In the absence of air or oxygen, it burns slow with a sooty greenish yellow flame liberating fluorine, which causes sporadic explosions.	Very stable. Non-sensitive to shock, percussion, heat, pressure, and friction. A secondary explosive. Requires a blasting cap or detonator for initiation.	Virtually chemically inert. Does not react with halogens even fluorine. Does not react with strong bases, acids, or strong oxidizers.	Water-white syrupy mobile liquid; Melting point: 12.9 Celsius; Boiling point: ~198 Celsius with decomposition beginning at 200 Celsius, rapid decomposition at temperatures over 267 Celsius. Insoluble in water, acetone, ether, THF, ethyl acetate, but slightly soluble

				in hexane, chloroform, methylene chloride, benzene, and moderately soluble in DMSO.

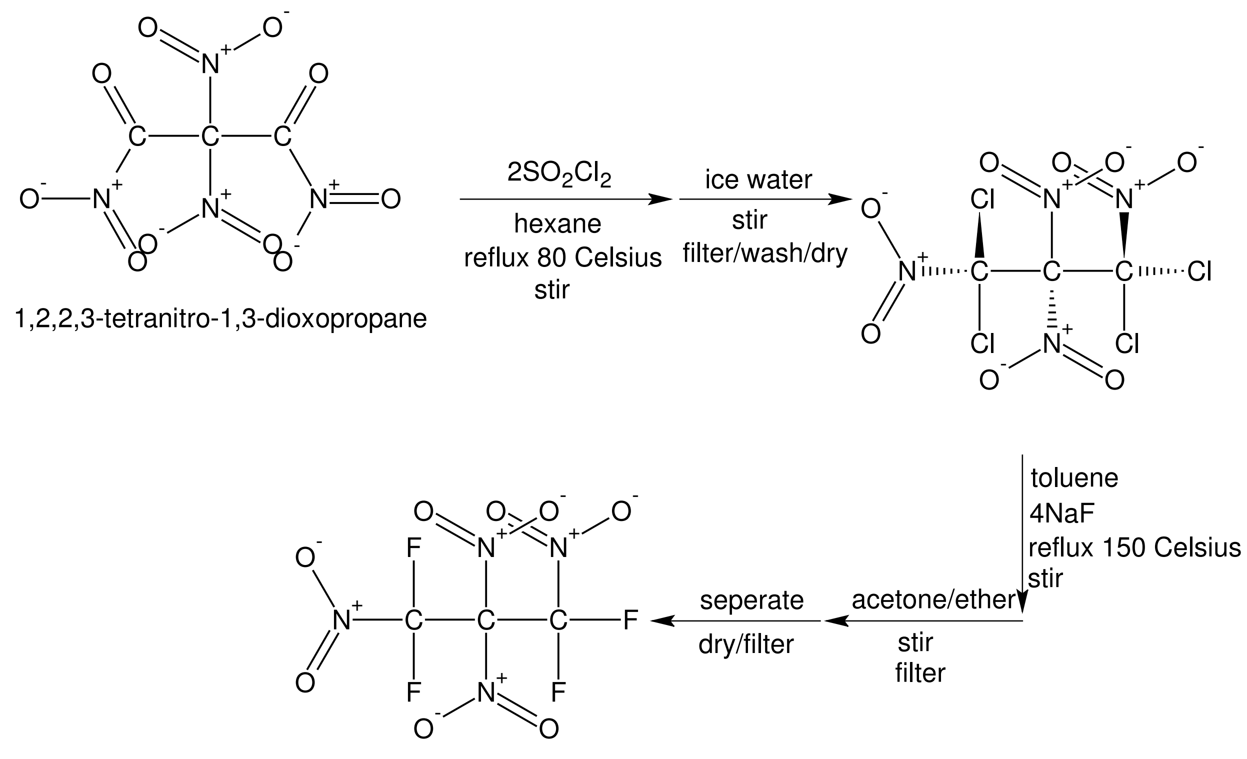
Estimated Compound summary: 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane is an awesome and powerful high energy plasticizer that can be used to make plastic explosives, high performance rocket fuels, and high performance gun propellants. For example, when 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane forms plastic explosives when admixed with solid high explosives and thoroughly mixed and then a little polypropylene glycol added and the mixture heated, and then cured forming a plumbers-putty like mass that can be molded into any desired explosive body for multiple military uses, especially improvised explosives. In another example, 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane forms either putty-like or rubber-like high performance rocket fuels with tremendous gas to weight ratio when it is mixed with a polyhydric nitrate such as nitroglycerine, and then added and mixed with nitrocellulose, and ammonium nitrate, and then cured with epoxy; these high performance rocket fuels can be used to propel missiles and rockets for various military operations. 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane also forms powerful high performance rocket fuels, when admixed with cyclic nitramines such as RDX or HMX, and cured with poly propylene glycol and epoxy. These high performance rocket fuels are very powerful and can propel heavy loads on rockets either for space launches/operations, or for long-range military rockets. 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane forms high performance gun propellants when admixed with nitrocellulose, and a double based oxidizer/reducer such as potassium nitrate and a flammable solid such as powdered aluminum, sulfur, or antimony trisulfide. These high performance gun propellants form dough-like masses that can be extruded into any desired grain size or shape for use in small arms weapons, and even high caliber guns such as battleship deck guns. 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane shows remarkable use in these three main categories i.e., plastic explosives, high performance rocket fuels, and high performance gun propellants.

Safety Info: Use caution when handling 1,2,2,3-tetranitro-1,3-dioxopropane, which is highly irritating to the eyes, nose, and throat; use maximum ventilation and avoid skin contact and inhalation. Extinguish all flames before using hexane, which is highly flammable. Wear gloves and avoid skin contact and inhalation of fumes when handling sulfuryl chloride, which is highly corrosive, and reacts violently with water, use caution. Use proper ventilation when handling toluene, and avoid skin contact and inhalation, as toluene is a known carcinogen. Be aware of acetone, which is highly volatile and flammable. Use maximum ventilation when handling ether, which is highly volatile and forms explosive mixtures with air. Also, perform the peroxide test on ether that has been in storage for prolonged periods of time to avoid possible explosions. Avoid inhalation of ether vapors, as the fumes are a narcotic in high concentrations.

Materials:

1. 197 grams of 1,2,2,3-tetranitro-1,3-dioxopropane (prepared in theoretical preparation 54)	6. 130 grams of sodium fluoride
2. 750 milliliters of dry hexane	7. 450 milliliters of acetone
3. 211 grams of sulfuryl chloride	8. 300 milliliters of diethyl ether
4. 1500 milliliters of a 10% sodium carbonate solution	9. 50 grams of anhydrous magnesium sulfate
5. 750 milliliters of dry toluene	

Reaction Equation:



1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane

Preparation:

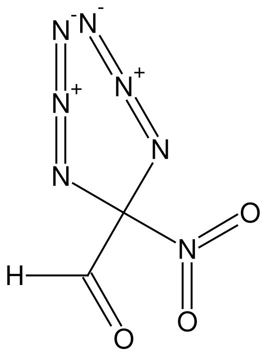
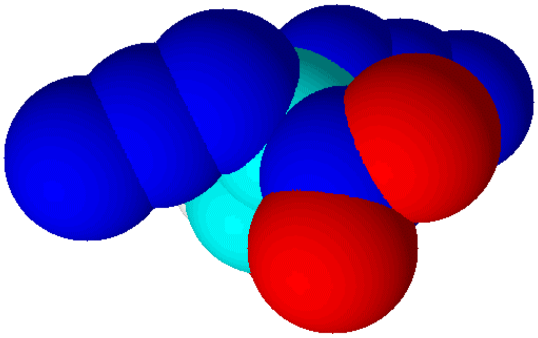
Step 1: preparation of 1,1,3,3-tetrachloro-1,2,2,3-tetranitropropane

Into a suitable sized 3-neck flask, sitting inside a suitable sized heating mantle, and this 3-neck flask equipped with motorized stirrer, addition funnel, and reflux condenser, place 197 grams of 1,2,2,3-tetranitro-1,3-dioxopropane (prepared in theoretical preparation 54), followed by 750 milliliters of dry hexane. Immediately thereafter, place into the addition funnel, 211 grams of sulfuryl chloride, and then turn on the motorized stirrer and begin to the sir the contents in the 3-neck flask. At the same time, turn on the heating mantle, and bring the temperature of the contents in the 3-neck flask to 80 Celsius under moderate reflux while stirring. Once the temperature of this reaction mixture reaches 80 Celsius, begin a slow drip of the sulfuryl chloride from the addition funnel into the reaction mixture while rapidly stirring the reaction mixture and maintaining its temperature at 80 Celsius under moderate reflux. Continue to slowly drip in the sulfuryl chloride from the addition funnel while rapidly stirring the reaction mixture and maintaining its temperature around 80 Celsius under reflux. Once all the sulfuryl chloride has been added, close the stopcock to the addition funnel, and continue to rapidly stir the reaction mixture under reflux of 80 Celsius for 4 hours. After 4 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, turn off the motorized stirrer, and then disassemble the apparatus, and then slowly and carefully pour the entire reaction mixture into a clean large beaker fitted with motorized stirrer (not magnetic stirrer), and filled with ice water. Once all the reaction mixture has been added to the ice water, rapidly blend this new mixture for 1 hour, adding more ice as needed during this 1 hour time period. After 1 hour, allow any remaining ice to melt, and then filter-off the insoluble mass using suction filtration, which will be the desired product, and then wash thoroughly with three 500-milliliter portions of a 10% sodium carbonate solution, followed by washing with six 750-milliliter portions of hot water, and then suction dry as best as possible. Thereafter, place this suction dried desired product of 1,1,3,3-tetrachloro-1,2,2,3-tetranitropropane into a dessicator over concentrated sulfuric acid for 7 days. After 7 days the dried product of 1,1,3,3-tetrachloro-1,2,2,3-tetranitropropane is ready for step 2.

Step 2: preparation of 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane

Into a suitable sized 3-neck flask, sitting inside a suitable sized heating mantle, and this 3-neck flask equipped with motorized stirrer, thermometer, and reflux condenser, place the dried product of 1,1,3,3-tetrachloro-1,2,2,3-tetranitropropane (obtained in step 1), followed by 750 milliliters of dry toluene, and then followed by 130 grams of sodium fluoride. Immediately thereafter, turn on the heating mantle, and bring the reaction mixture to strong reflux by heating to 150 Celsius with constant stirring. When the reaction mixture reaches 150 Celsius under rapid stirring, continue to heat and stir this reaction mixture at 150 Celsius under strong reflux for 6 hours. After 6 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and the pour the entire reaction mixture into a clean large beaker, equipped with motorized stirrer (not magnetic stirrer). Now, pour into this beaker a solvent mixture obtained by adding and dissolving 450 milliliters of acetone into 300 milliliters of diethyl ether, and then blend this new mixture for 1 hour. After 1 hour, turn off the motorized stirrer, and then filter-off all the insoluble by-product material using suction filtration. Now, take the filtered mixture, and allow to stand for 1 hour—during this time, the desired product will separate out forming a lower liquid layer. Now, use a separatory funnel to collect this lower liquid layer by carefully draining it off, and then once this lower liquid layer has been recovered, place it into a clean beaker, and then add in 50 grams of anhydrous magnesium sulfate, and then stir for 1 hour. After 1 hour, stop stirring, and then filter-off the magnesium sulfate using suction filtration, and then take the desired syrupy liquid product of 1,1,3,3-tetrafluoro-1,2,2,3-tetranitropropane and store it in an amber glass bottle in a cool dry place until use.

Theoretical Preparation 56: diazido(nitro)acetaldehyde; DA-NA; DA-nitro-A

 diazido(nitro)acetaldehyde	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_2HN_7O_3$
Formula Weight = 171.07444
Composition = C(14.04%) H(0.59%) N(57.31%) O(28.06%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 171.014087 Da
Nominal Mass = 171 Da
Average Mass = 171.0744 Da

Molecular Information:

Detonation velocity (estimated by	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

monoisotopic mass and index of refraction):				
6820	Non-flammable; will not burn even when heated and ignited, but forms highly flammable mixtures with powdered metals and strong reducing agents.	Stable; is non-sensitive to shock, percussion, heat, pressure, and friction. A secondary explosive. Requires blasting cap or detonator for proper initiation.	Is easily oxidized by strong oxidizing agents; may explode if heated with potassium permanganate. Does not react with the halogens, except for fluorine causing the compound to ignite in flames. Reacts with strong bases in solution forming aldol condensation products.	White granules or powder. Melting point 67 Celsius; Boiling point 209 Celsius under reduced pressure. Insoluble in water, and most organic solvents.

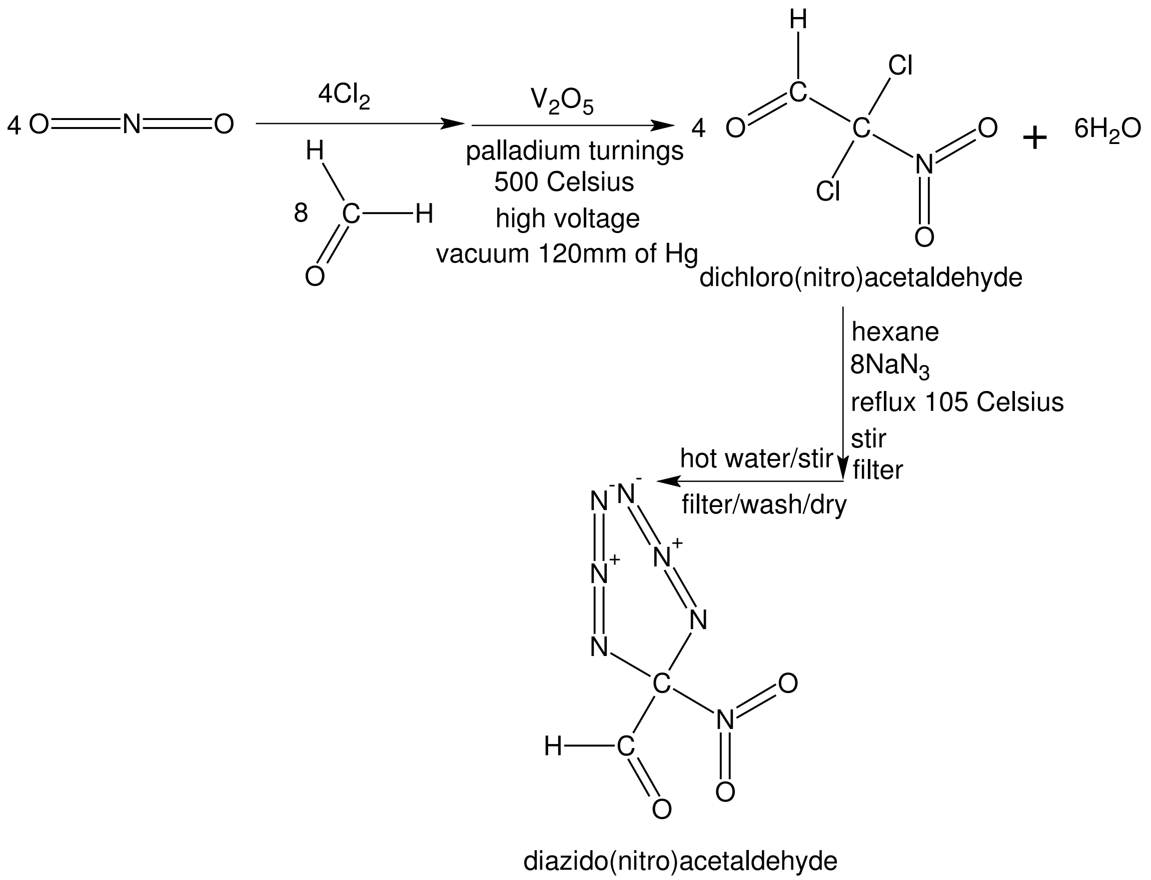
Estimated Compound summary: diazido(nitro)acetaldehyde is new and unique aldehyde containing high explosive with satisfactory properties. diazido(nitro)acetaldehyde has a relatively low melting point making it well suitable for melting and casting into shape charges, or for pouring into mortar shells, artillery shells, bomb casings, or for pouring and casting into missile warheads, or any desired shape and/or size for multiple commercial and military operations. diazido(nitro)acetaldehyde can be used to make high performance solid rocket propellants when mixed with nitrocellulose and a high energy plasticizer and cured, and these solid rocket fuels have tremendous thrust to weight ration making them well suitable for making any desired sized rocket for multiple commercial and military operations. diazido(nitro)acetaldehyde can also be used to make high performance gun propellants when admixed with a polyhydric nitrate compound such as nitroglycerine and then cured with epoxy or nitrocellulose. The gun propellants produce tremendous gas pressures making them well suitable for manufacturing small arms ammunitions, propellants for tank-fired projectiles, and for making propellants for large caliber guns such as battleship deck guns. diazido(nitro)acetaldehyde is considered non-flammable, but when it is mixed in the right proportions with powdered metals or strong reducing agents it forms highly flammable mixtures that can be used in pyrotechnics for various operations and uses. Note: diazido(nitro)acetaldehyde when mixed with powdered metals such as powdered aluminum or magnesium and cured with epoxy form powerful solid rocket fuels.

Safety Info: Use maximum ventilation and avoid inhalation when handling nitrogen dioxide, which is toxic. Use maximum ventilation and avoid inhalation of formaldehyde gas, which is toxic. Use maximum ventilation when handling chlorine gas and avoid inhalation, as chlorine is toxic. Extinguish all fames before using hexane, which is volatile and highly flammable. Wear gloves and avoid skin contact and inhalation of dust and ingestion of sodium azide, which is very toxic.

Materials:

1. 140 grams of vanadium pentoxide	6. 171 grams of chlorine gas
2. 50 grams of palladium turnings	7. 50 grams of anhydrous magnesium sulfate
3. Argon gas from tank	8. 1200 milliliters of dry hexane
4. 111 grams of nitrogen dioxide gas	9. 331 grams of sodium azide
5. 145 grams of formaldehyde gas	

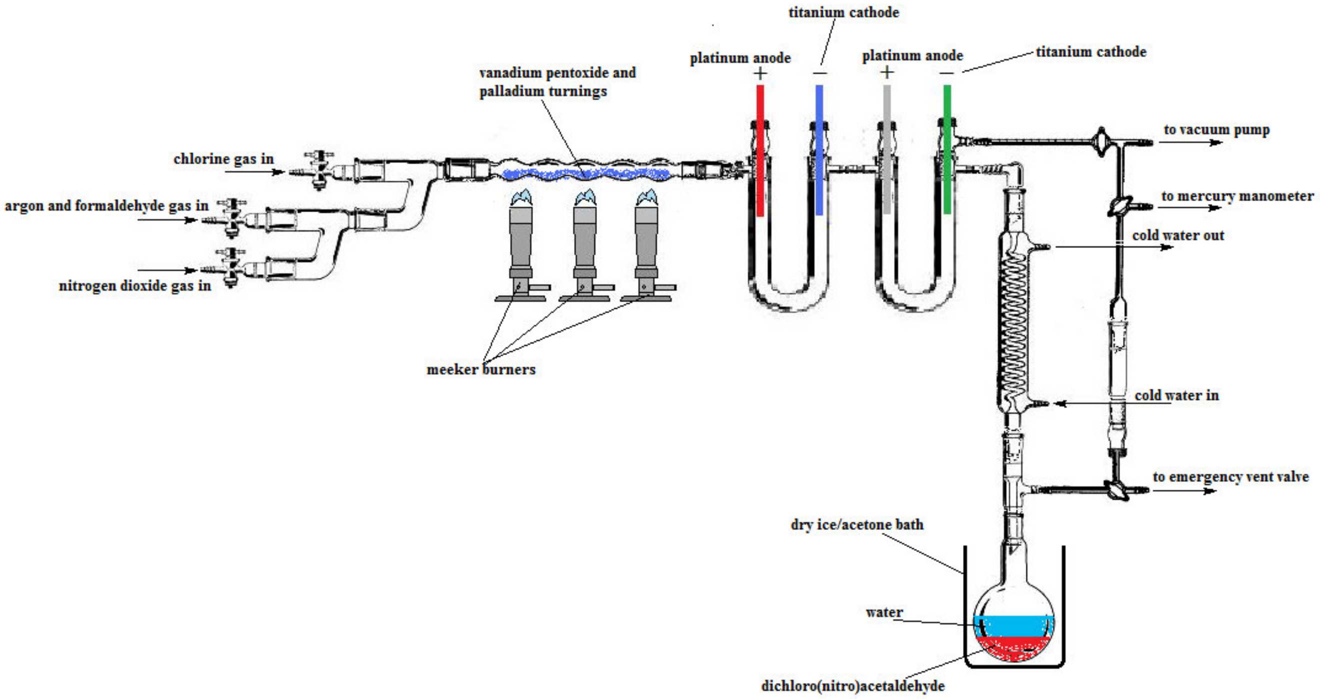
Reaction Equation:



Preparation:

Step 1: preparation of dichloro(nitro)acetaldehyde

First, into the bulb tube as illustrated place 140 grams of vanadium pentoxide, and 50 grams of palladium turnings, and then shake the bulb tube to evenly distribute the vanadium pentoxide and palladium turnings in the bulb tube as illustrated. Thereafter, turn on the meeker burners and heat this bulb tube to 500 Celsius. Thereafter, begin an argon gas purge by passing dry argon gas into the apparatus at a flow rate of 500 mg per/minute, and then attach the red positive clamps to the positive anodes, and attach the black negative clamps to the negative cathodes, as illustrated, and then turn on the high voltage power supply as illustrated. Note: the argon will glow with a brilliant florescent light in the high voltage U-tubes. Thereafter, turn on the vacuum pump and bring the pressure inside the apparatus to 120mm of mercury. Once the pressure inside the apparatus is at 120mm of mercury, and the temperature of the ingredients in the bulb tube reaches 500 Celsius, begin to pass into the apparatus dry nitrogen dioxide gas at a flow rate of 45 mg per/minute, and then pass into the apparatus dry formaldehyde gas at a flow rate of 52 mg per/minute, and then pass into the apparatus dry chlorine gas at a flow rate of 75 mg per/minute while heating the bulb tube to 500 Celsius under a vacuum of 120mm of mercury. Continue to purge the apparatus with argon gas, and continue to pass into the apparatus nitrogen dioxide gas until 111 grams of nitrogen dioxide gas has been added and 145 grams of formaldehyde gas has been added, and 171 grams of chlorine gas has been added. Once all the required nitrogen dioxide gas, formaldehyde gas, and chlorine gas has been added, turn off the high voltage power supply, and then turn off the meeker burners, and then turn off the vacuum pump and allow the pressure inside the apparatus to reach STP, and remove the dry ice/acetone bath as illustrated below, and allow the reaction products to warm to room temperature. Thereafter, disassemble the apparatus, and then pour the reaction products into a separatory funnel, and then drain off the bottom product layer, which will be the desired dichloro(nitro)acetaldehyde. Finely, pour this liquid product layer into a clean beaker, and then add in 50 grams of anhydrous magnesium sulfate, to absorb moisture, and then stir for 5 minutes. Thereafter, suction filter the product mixture to remove the insoluble magnesium sulfate, and then recover the liquid product and save for step 2.

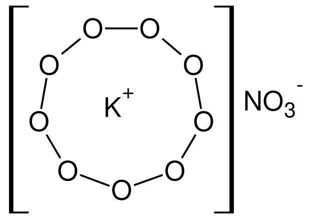
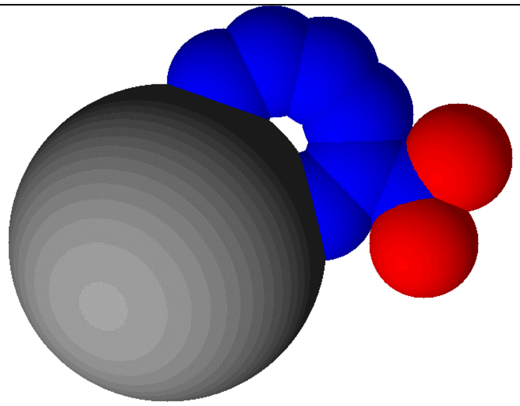


Advanced apparatus for preparing the intermediate dichloro(nitro)acetaldehyde.

Step 2: preparation of diazido(nitro)acetaldehyde

Into a standard reflux apparatus utilizing a 3-neck flask sitting inside a standard round bottom heating mantle, and equipped with motorized stirrer, bulb condenser, and thermometer, place the liquid product of dichloro(nitro)acetaldehyde in to the 3-neck flask, and pour in 1200 milliliters of dry hexane, followed by 331 grams of sodium azide. Now, turn on the heating mantle, and reflux this reaction mixture to 105 Celsius under rapid stirring for 12 hours. After 12 hours, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter-off all the insoluble solids (including the by-products) using suction filtration, and then suction dry as best as possible. Now, take the filtered-off insoluble suction dried mass, and place it into a clean beaker sitting on top of a hot plate equipped with motorized stirrer (not magnetic stirrer), and then add in 2500 milliliters of tap water, and then turn on the hot plate and heat to 90 Celsius under rapid stirring. During this operation the by-products, mainly sodium chloride, will dissolve in the water, leaving the desired product insoluble in the water mixture. Continue to heat this water solution for 4 hours at 90 Celsius under rapid stirring. After 4 hours, turn off the hot plate, and then filter-off the insoluble desired product while the water mixture is still hot using suction filtration, and then wash with three 750-milliliter portions of hot water, and then suction dry as best as possible. Finely, take this suction dried desired product and place it onto a shallow pan or tray and allow it to air-dry for 7 days. After 7 days, scrap up the dried desired product and store in an amber glass bottle in a cool dry place until use.

Theoretical Preparation 57: potassium nonoxonane nitrate; Trioxy potassium nitrate; nitrate of potassium nonoxonane; KON

 potassium nonoxonane nitrate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = KNO_{12}
Formula Weight = 245
Composition = O(78.36%) K(15.95%) N(5.7%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = 2.0009 estimated
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 181.250098 Da
Nominal Mass = 181.09 Da
Average Mass = 181.4580 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
4670	Non-Flammable; forms explosive mixtures with reducing agents mainly powdered metals.	Non-sensitive to shock, pressure, percussion, or friction. Moderate sensitivity to heat; begins to decompose when rapidly heated to ~150 Celsius. A secondary explosive. Requires a blasting cap or detonator for initiation.	Generally moderately reactive. Reacts violently with the halogens, especially fluorine resulting in explosion. Does not react with ozone gas or potassium permanganate. Also, does not react with strong oxidizers. Reacts with strong bases when dry and heated, but not while in solution.	Light blue cubic crystals, or white powder or granules; Melting point: 104 Celsius depending on purity; Boiling point: decomposes when heated to 167 Celsius, but decomposes when rapidly heated to 150 Celsius, often explosively with evolution of ozone and nitrogen

			Decomposes in concentrated sulfuric acid, and may explode when mixed with perchloric acid. Decomposes when mixed with strong acids.	dioxide gases. Moderately soluble in water, and slightly soluble in alcohol. Very soluble in sucrose solution (from which it can be recrystallized from forming beautiful blue transparent cubic crystals). Insoluble in most organic solvents. Soluble in liquid ammonia, and liquid sulfur dioxide.

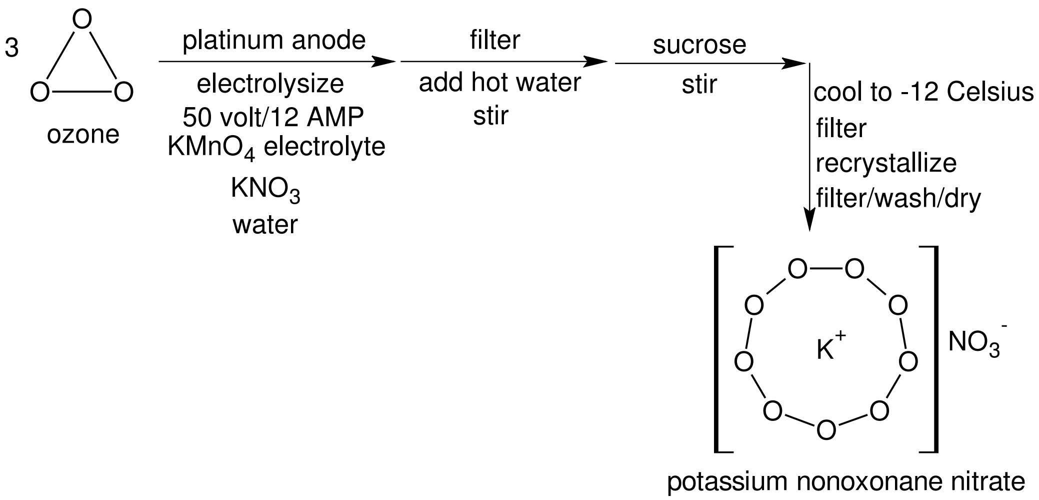
Estimated Compound summary: potassium nonoxonane nitrate is a one of kind radical explosive containing a 9 oxygen ring with a positively charged potassium ion in the center of the oxygen ring. This type of explosive is part of a whole new revolution of high explosives that contain large atomic rings that are capable of trapping positively charged ions. In this regard, the positively charged ions can range from simple ions like potassium or sodium, but more complex positively charged ions such as the ammonium ion, and others. Potassium nonoxonane nitrate has interesting physical properties with a remarkable higher then calculated melting point. It can be melted down, and poured into molds for making shape charges, but its low detonating velocity sort of defeats this purpose; however, potassium nonoxonane nitrate can be melted down and poured into shell casings for making mortar bombs, artillery shells, and for tank fired projectiles for making anti-personnel fragmenting munitions (where high power of detonation is not recommended). One of the major uses of potassium nonoxonane nitrate is in the rocket and propellant field. It forms a powerful solid rocket fuel when mixed with powdered aluminum, a little nitrocellulose, and then cured with epoxy. This propellant is easily ignited and burns with a tremendous thrust to weight ratio making it well suitable for a variety of commercial and military applications. Other high performance rocket propellants can be made using potassium nonoxonane nitrate when mixed with strong reducing agents and cured with epoxy or a high-energy plasticizer. potassium nonoxonane nitrate can also be used in making high performance gun propellants when admixed with nitrocellulose, and any desired strong reducing agent and then cured with a high energy plasticizer or epoxy. In short, the use of potassium nonoxonane nitrate in making explosive munitions is very limited, and its use would most likely be in the rocket and propellant industry where it shows remarkable promise.

Safety Info: Use care when handling potassium permanganate and avoid contact with combustible materials as potassium permanganate is a powerful oxidizing agent. Use maximum ventilation when handling ozone gas, which is highly irritating to the nose, throat, and eyes. Use care when handling ozone when combustible materials are present. Extinguish all sources of ignition when handling acetone, which is highly volatile and flammable.

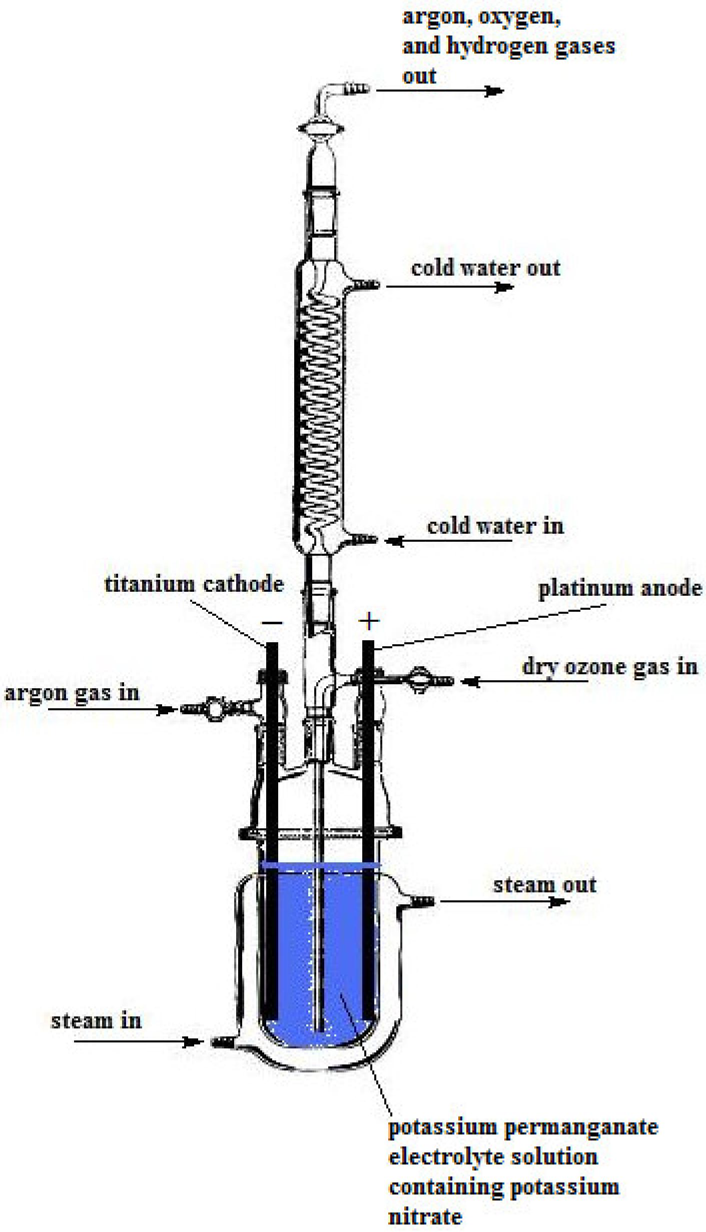
Materials:

1. 150 grams of potassium permanganate	4. 1500 grams of granulated sucrose
2. 72 grams of potassium nitrate	5. 750 milliliters of acetone
3. 103 grams of ozone gas	

Reaction Equation:

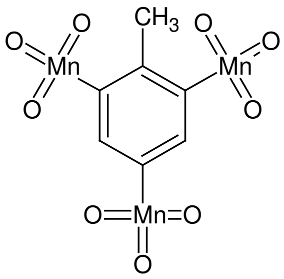
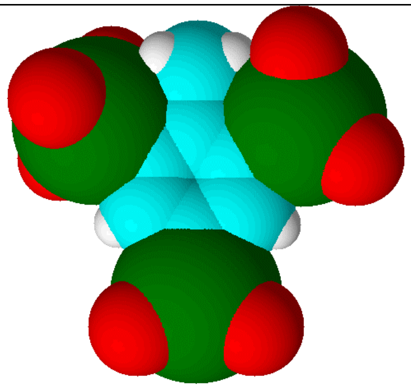


Preparation: Into an apparatus as illustrated below, pour in 1500 milliliters of hot water, and then add in 150 grams of potassium permanganate, followed by 72 grams of potassium nitrate, and then manually stir this mixture until all solids have dissolved. Thereafter, assemble the apparatus as illustrated, begin to purge the apparatus with argon gas, and then attach the electrical power clamps to their appropriate electrodes as illustrated (black clamp on the cathode, and red clamp on the anode). Then, begin to pass in hot steam pre-heated using any desired means (not illustrated) through the outer jacket of the reaction vessel. Now, wait about 15 minutes to allow the steam to heat the electrolyte reaction mixture, and then begin to pass in ozone gas at a flow rate of 15 mg per/minute, and then immediately thereafter, turn on your power supply to 50 volt/12AMP DC current, and then continue to pass in this ozone gas until 103 grams of ozone gas has been added. Now, once all the ozone gas has been added, immediately turn off the power supply, turn off the steam source, and turn off the argon purge. Then allow the electrolysized reaction mixture to cool to room temperature. Once the electrolysized reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter this reaction mixture to recover the precipitated manganese dioxide by-product using suction filtration. After filtration, take the filtered reaction mixture and place it into a large beaker sitting on top of a magnetic stirrer, and then add in 750 milliliters of pre-heated water (using any desired means to around 60 Celsius), and then stir this diluted filtered reaction mixture for about 15 minutes using a Teflon stir bar. Now, add in all at once 1500 grams of granulated sucrose, and then continue to stir this new mixture until the sugar dissolves. Once the sugar dissolves, allow this new mixture to cool to room temperature, and then place it into an ice/salt bath and chill to around –12 Celsius. Once the temperature of this sugar reaction mixture reaches –12 Celsius, hold at this temperature for 4 hours. Note: during this 4 hour time period, another by-product from the original electrochemical reaction will precipitate (which will be potassium hydroxide). After 4 hours, filter-off the precipitated potassium hydroxide by-product using suction filtration, and then place the filtered sugar mixture into a larger clean beaker sitting on top of a hot plate, and then begin the recrystallization process by bringing the mixture to a gentle boil (add boiling stones to aid in the gentle boiling process), and boil-off some of the water until tiny little crystals begin to appear on the surface of the sugar mixture. Once tiny little crystals begin to form, turn off the heat source, and allow the sugar mixture to cool. Note: during this cool down period, brilliant blue transparent cubic crystals of the desired product potassium nonoxonane nitrate will form. Now, once the sugar mixture has cooled to around 40 Celsius, filter-off the precipitated crystals of the desired product of potassium nonoxonane nitrate using suction filtration (utilizing a Buchner funnel), and then take the filtered sugar mixture and place it back onto the hot plate and bring it to a gentle boil, to boil-off more water until tiny crystals begin to form on the surface of the sugar solution as before. Once this happens, turn off the heat source, and allow the sugar solution to cool. Note: during this cool down period, more brilliant blue transparent cubic crystals of the desired product potassium nonoxonane nitrate will form. Now, once the sugar mixture has cooled to around 40 Celsius as before, filter-off the precipitated crystals of the desired product of potassium nonoxonane nitrate using suction filtration (using the same Buchner funnel), and then take the filtered sugar mixture and place it back onto the hot plate and bring it to a gentle boil, to boil-off more water until tiny crystals begin to form on the surface of the sugar solution as before—repeat this process two more times using the same Buchner funnel to collect the crystallized filtered-off crystals of potassium nonoxonane nitrate. After repeating the heating and cooling process two more times, most of the desired product of potassium nonoxonane nitrate would have been collected. Finally, to all the collected crystals of potassium nonoxonane nitrate in the Buchner funnel, wash these crystals with three 250-milliliter portions of acetone using suction filtration, and then suction dry as best as possible. Thereafter, place the washed crystals into an oven and gently heat to 60 Celsius for 24 hours. After 24 hours, turn off the oven, and allow the dried desired product to cool to room temperature before removing. Once the dried product has cooled to room temperature, recover the desired product of potassium nonoxonane nitrate, and then store in an amber glass bottle in a cool dry place until use.


argon, oxygen, and hydrogen gases out
cold water out
cold water in
titanium cathode
platinum anode
−
+
dry ozone gas in
argon gas in
steam out
steam in
potassium permanganate electrolyte solution containing potassium nitrate

Setup for preparing potassium nonoxonane nitrate.

Theoretical Preparation 58: (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide; Tri-permanganic toluene; TTP

 (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_7H_5Mn_3O_9$
Formula Weight = 397.923335
Composition = C(21.13%) H(1.27%) Mn(41.42%) O(36.19%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = Not available
Nominal Mass = Not available
Average Mass = Not available

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
4690	Non-flammable; will not burn even when mixed with reducing agents and ignited with a flame.	Very stable; non-sensitive to heat, pressure, percussion, and friction A secondary explosive. Requires a blasting cap or detonator for initiation.	Very stable to chemical attack. Virtually inert to all chemical attacks.	White to colorless crystalline powder. Melting point: 209 Celsius; boiling point: decomposes slowly when heated to 490 Celsius, with rapid decomposition when heated at 512 Celsius. Insoluble in water, and all known solvents. Even insoluble in

				liquid ammonia and liquid sulfur dioxide.

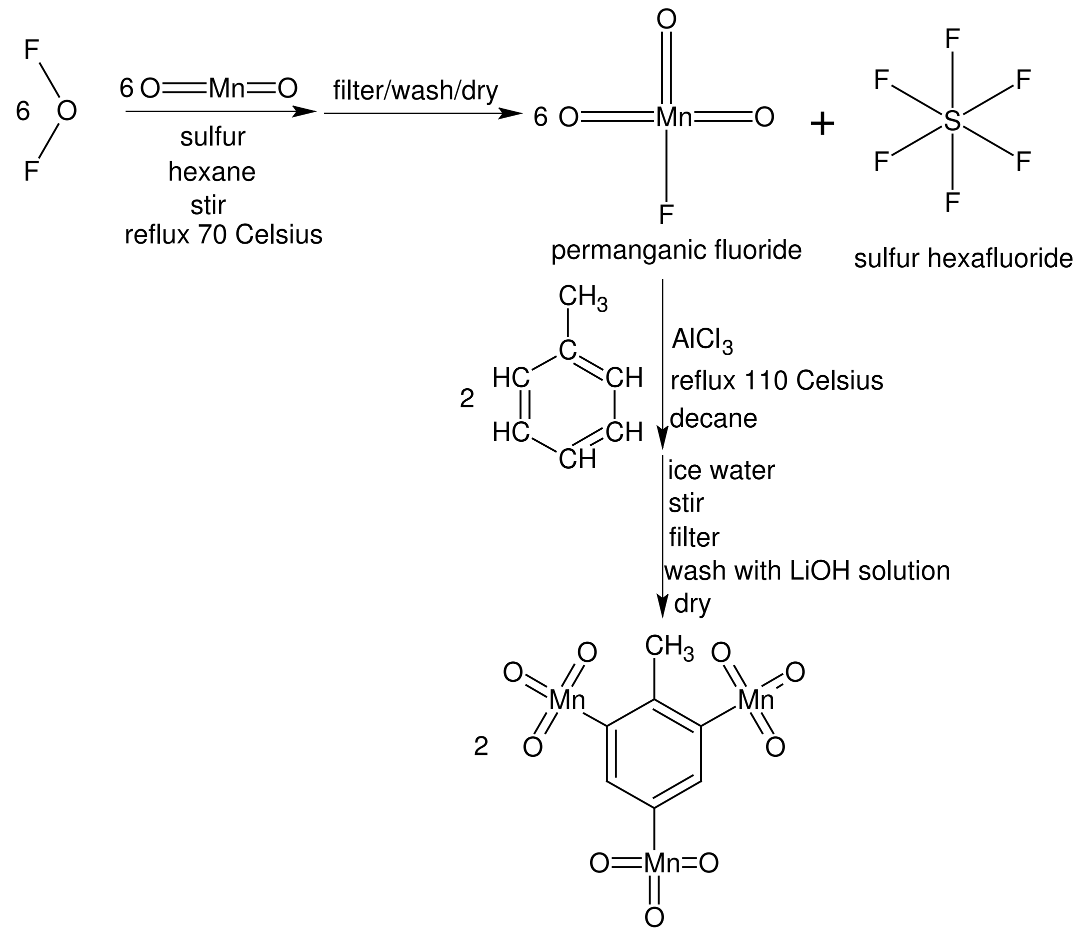
Estimated Compound summary: (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide is a unique and remarkable new high explosive with very interesting properties. It is insoluble in all known solvents, and is very stable. It has outstanding chemical inertness and does not even react with fluorine gas. (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide requires a significant detonator utilizing a base charge of RDX or HMX for proper initiation. Note: regular sized blasting caps, or small detonators will not properly initiate (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide. (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide has a relatively high melting point, making it suitable for space based weapons. The explosive can also be melted down, and poured into molds for shape charges, for pouring into mortar or artillery shells, or for pouring into bomb casings and then allowed to cool. As previously mentioned, these munitions need large detonators for proper initiation. (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide has no use in making gun propellants, or solid rocket fuels, or in making pyrotechnic compositions.

Safety Info: Extinguish all flames before using hexane, which is highly flammable. Use proper ventilation when handling oxygen difluoride, and avoid inhalation of the fumes. Wear gloves when handling anhydrous aluminum chloride, and avoid contact with moisture. Wear gloves when handling lithium hydroxide solution, as it can cause skin irritation. Use proper ventilation when handling toluene, which is a known carcinogen, avoid inhalation and skin contact.

Materials:

1. 750 milliliters of hexane	6. 100 grams of anhydrous aluminum chloride
2. 102 grams of manganese dioxide	7. 750 grams of decane
3. Argon gas from tank	8. 42 grams of toluene
4. 97 grams of oxygen difluoride	9. 750 milliliters of a 25% lithium hydroxide solution
5. 750 milliliters of fresh methylene chloride	10. 4 grams of flours of sulfur

Reaction Equation:

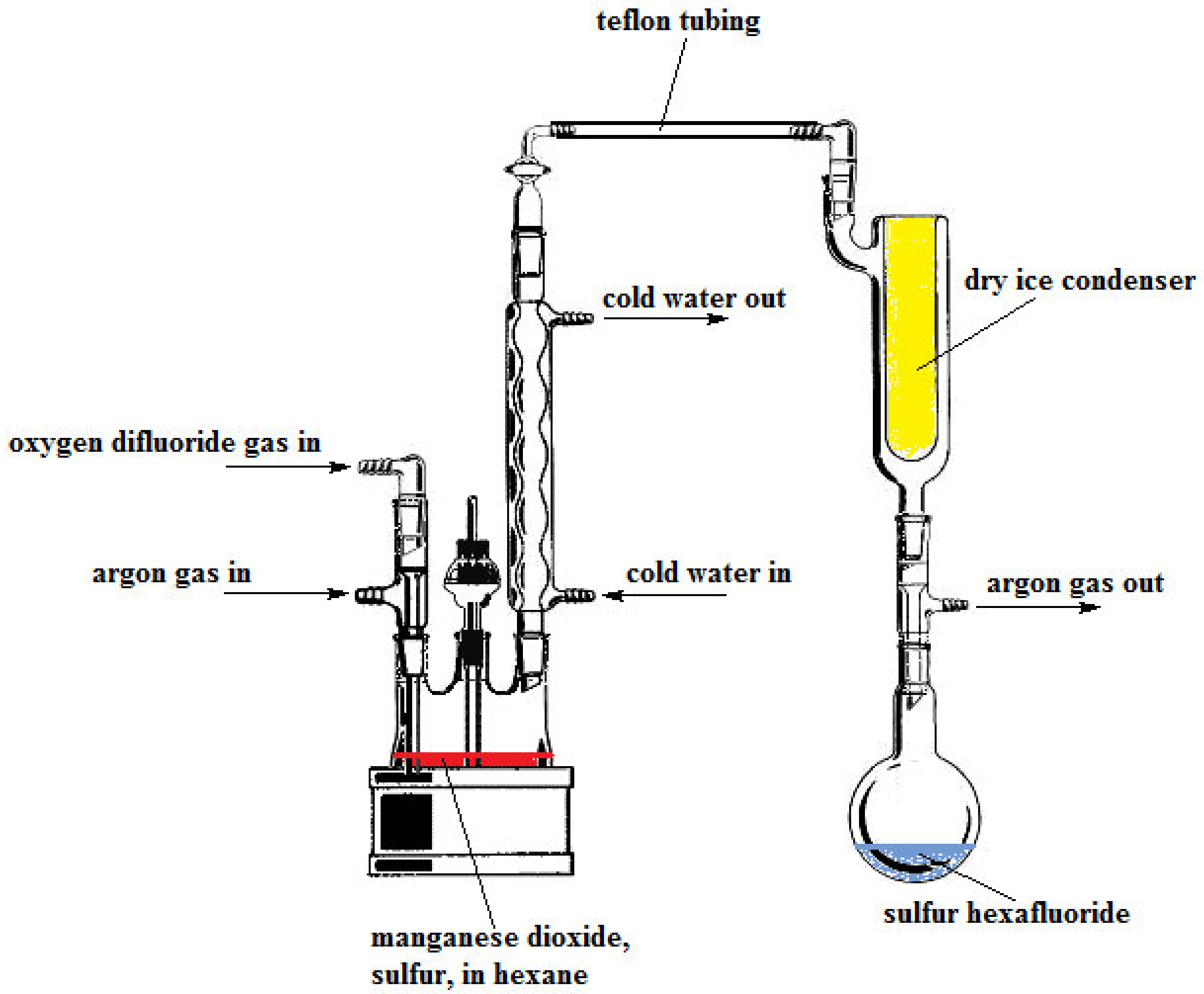


(2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide

Preparation:

Step 1: The preparation of permanganic fluoride

Into an apparatus as illustrated below, pour in 750 milliliters of hexane followed by 102 grams of manganese dioxide, and then add in 4 grams of flours of sulfur, and then begin to stir this mixture. Then turn on the heating mantle and bring the contents in the reaction flask to a gentle reflux by heating to 70 Celsius with constant stirring. Now, begin an argon gas purge at a flow rate of 50 mg per/minute, and then immediately thereafter, begin to bubble into the reaction mixture oxygen difluoride from a tank at a flow rate of 75 mg per/minute while rapidly stirring the reaction mixture and maintaining it's reflux at 70 Celsius. Continue to bubble into the reaction mixture oxygen difluoride gas until a total of 97 grams of the gas has been bubbled into the reaction mixture. After the addition of the oxygen difluoride gas, continue to purge the apparatus with argon gas, and continue to reflux the reaction mixture under vigorous stirring for about 4 hours. Note: during the reaction, the heavy gas sulfur hexafluoride will slowly pass over and will condense into a liquid by means of a dry ice condenser. After 4 hours, turn off the argon purge, turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during the cool down period, continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, disassemble the apparatus, and then filter-off the insoluble desired product of permanganic fluoride using vacuum filtration, and then wash with three 250-milliliter portions of fresh methylene chloride, and then vacuum dry as best as possible. Finally, place the suction dried washed desired product of permanganic fluoride into a dessicator over sodium hydride for 7 days, and then save for step 2.

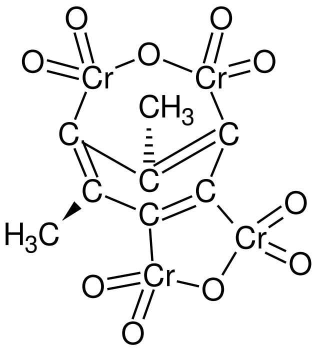


Apparatus for the preparation of permanganic fluoride. The sulfur hexafluoride collects in the bottom receiver flask as a cold liquid, and can be discarded if desired. Note: due to the inertness of the sulfur hexafluoride, it cannot be recycled.

Step 2: preparation of (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide

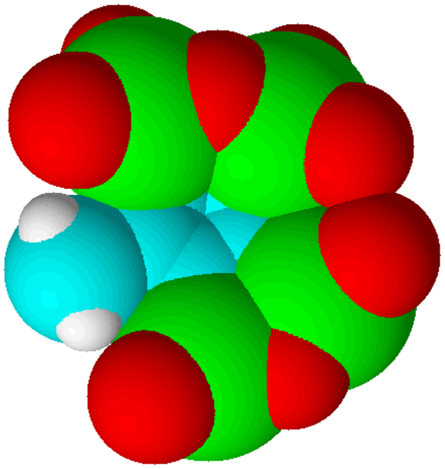
Into a standard reflux apparatus, equipped with motorized stirrer, and reflux condenser (open at the top), place 750 grams of decane, followed by 100 grams of anhydrous aluminum chloride, followed by 42 grams of toluene, and then turn on the heating mantle and begin to heat the reaction mixture to 110 Celsius under rapid stirring. Once the reaction mixture reaches a temperature of 110 Celsius, begin to add in, through the top of the reflux condenser (so that the additions fall straight down the reflux condenser and into the reaction mixture), in small portions at a time, the permanganic fluoride prepared in step1. During the addition of the permanganic fluoride continue to heat the reaction mixture at 110 Celsius under rapid stirring. Now, once all the permanganic fluoride has been added to the reaction mixture, continue to heat the reaction mixture at 110 Celsius under rapid stirring for 2 hours. After 2 hours, turn off the heating mantle, and allow the reaction mixtures temperature to warm to 80 Celsius, and during this cool down period continue to rapidly blend the reaction mixture. Once the reaction mixture has cooled to around 80 Celsius, quickly disassemble the apparatus, and then pour the entire reaction mixture, carefully and slowly, into 2000 milliliters of ice water contained in a 4-liter beaker. Thereafter, stir this aqueous mixture for 4 hours using any desired stirring means, adding more ice as needed. After 4 hours, filter-off the insoluble desired product of (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide using suction filtration, and then suction dry as best as possible. Now, wash this filtered-off desired product with three 250-milliliter portions of a 25% lithium hydroxide solution, followed by washing with six 500-milliliter portions of hot water, and then suction dry as best as possible. Finely, place the washed suction dried desired mass of (2-methylbenzene-1,3,5-triyl)(tris)(permanganic) nonaoxide into an oven and bake at 105 Celsius for 6 hours. After 6 hours, turnoff the oven, and allow the dried desired product to cool to room temperature before removing. Once the desired product has cooled to room temperature, remove it from the oven and store it in a glass jar until use.

Theoretical Preparation 59: 7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo [6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide; Bicyclo-dichromate-meta-cresol; Chromsplode Ajax; AJAX-109



7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo [6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide

Expanded structure



3D structure

Molecular data:

Molecular Formula = $C_8H_6Cr_4O_{10}$
Formula Weight = 470.1204176
Composition = C(20.44%) H(1.29%) Cr(44.24%) O(34.03%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = 2.009 ± 0.0099
Surface Tension = Not available
Density = 3.098 ± 0.034 g/cm^3
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 469.766922 Da
Nominal Mass = 470 Da
Average Mass = 470.1204 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7087	Non-flammable; forms explosive mixtures with reducing agents.	Very stable; stable towards shock, percussion, heat, pressure, and friction; however, mixtures with hypophosphites are shock sensitive, and mixtures with chromates and	Generally stable to chemical attack, does not react with fluorine or the other halogens and does not react with acids or bases under normal conditions; however, can be chlorinated or	Forms beautiful yellowish cubic crystals with a melting point of 236 Celsius. Decomposes when heated to above 670 Celsius. Insoluble in water, and all

		powdered boron explodes when touched by a spark or flame. Normally a secondary explosive. Under most conditions requires a blasting cap or detonator for initiation.	brominated in the presence ferric chloride forming chlorine or bromine derivatives (neither of which have any economical value).	known solvents.

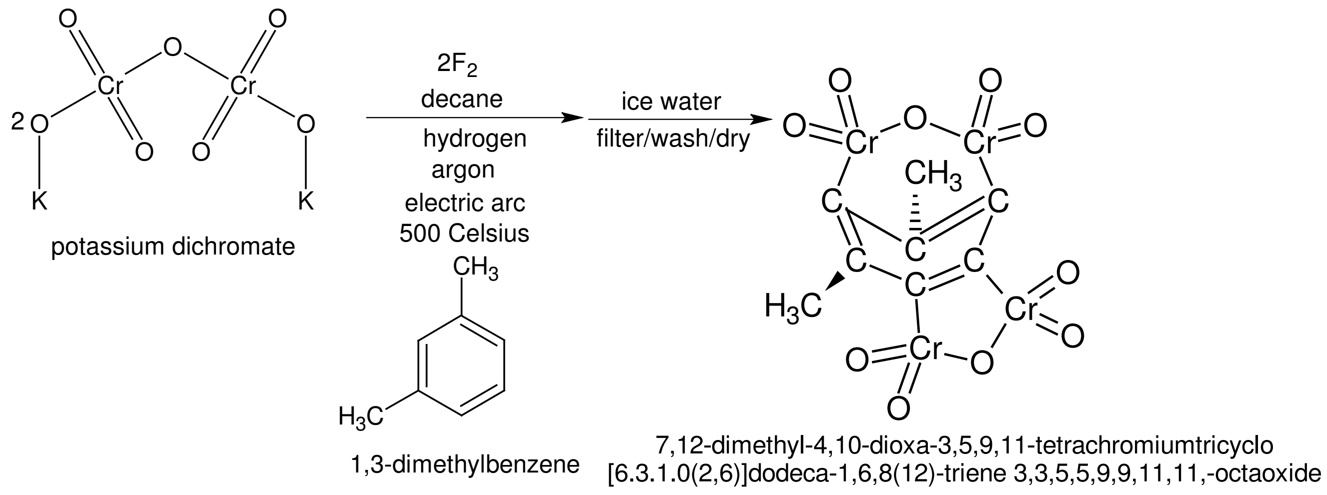
Estimated Compound summary: 7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo [6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide is a spectacular chromium based high explosive with an amazing higher then expected detonating velocity. Because of it's high melting point and stability, it can be melted down and poured in to shells for making mortar bombs, artillery shells, gravity bombs, laser guided bombs, or poured into any desired molds for making rockets and missile warheads, and then allowed to cure. This particular high explosive is an amazing dichromate based explosive and a one of a kind in its field. 7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo [6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide can be used in making high performance gun propellants and high performance rocket propellants when mixed with reducing agents such as powdered metals, and then cured with epoxy, the latter primarily for making high performance rocket fuels. For making high performance gun propellants, 7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo[6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide can be slurried with liquid explosives such as nitroglycerine or other polyhydric nitrates, nitrocellulose added, and then extruded under high pressure into strands or pellets and then allowed to cure. These high performance gun propellants produce moderate to high chamber pressures primarily suitable for small arms weapons and not for heavy or large bore guns as seen with tanks, field guns, deck guns, or battleship guns. 7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo [6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide has some irregular properties that make it suitable for making a whole new era of pyrotechnic compositions for many uses, and the combinations of this explosive with reducing agents is practically endless forming an arsenal of cool pyrotechnic effects. Further investigation is warranted.

Safety Info: This entire reaction process is very dangerous as high voltage electricity and fluorine gas are being used. Use extreme caution when carrying out this process. Fluorine gas is highly reactive and dangerous, and explodes in contact with many organic substances, use extreme caution and avoid inhalation of fluorine gas. Note: carryout this process in a well ventilated area as the high voltage current will create a magnetic field and will turn oxygen in toxic ozone gas. Extinguish all flames, and use maximum ventilation when handling diethyl ether as is forms explosive mixtures with air. Also, use the usual cautions when handling hydrogen gas, which also forms explosive mixtures with air.

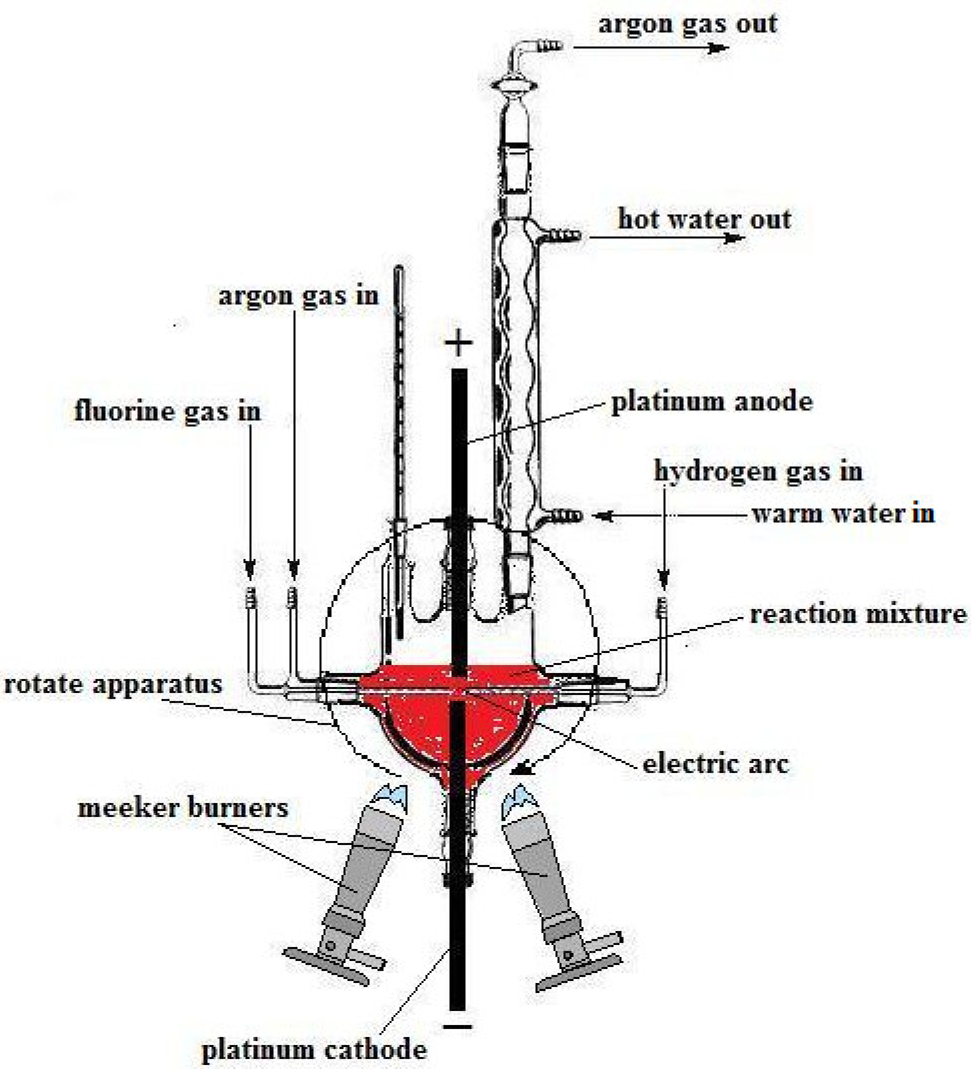
Materials:

1. 750 milliliters of n-decane	4. 1 gram of hydrogen gas
2. 126 grams of potassium dichromate	5. 16.2 grams of fluorine gas
3. 23 grams of meta-cresol	6. 750 milliliters of dry fresh diethyl ether

Reaction Equation:

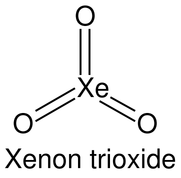
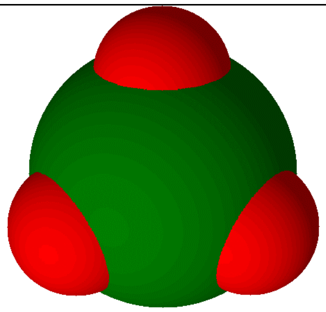

2
potassium dichromate
$2F_2$
decane
hydrogen
argon
electric arc
500 Celsius
1,3-dimethylbenzene
ice water
filter/wash/dry
7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo
[6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide

Preparation: Into a special apparatus as illustrated below, fill the reaction chamber with 750 milliliters of n-decane, and then add in 126 grams of potassium dichromate, followed by 23 grams of meta-cresol, then assemble the apparatus as illustrated below, then attach the black negative clamp to the negative cathode, and then attach the red positive clamp to the positive anode, and using meeker burners, or any desired heating method, heat the reaction chamber to 500 Celsius, and allow the n-decane to vigorously boil. Once the n-decane begins to vigorously boil, begin to pass into the apparatus dry argon gas at a flow rate 0.500 mg per/minute, and then wait 10 minutes to flush all the air out of the system. After about 10 minutes, begin to pass in dry hydrogen gas at a flow rate of 0.05 mg per/minute, and at the same time, begin to pass into the apparatus, dry fluorine gas at a flow rate of 0.81 mg per/minute. Now, immediately after you begin passing into the apparatus hydrogen and fluorine gasses, turn on your power supply at 50,000 volt at 0.1 amps DC. Warning! Do not touch both electrodes at the same time otherwise you will get the shock of your life! Once the power has been turned on, continue to pass in hydrogen gas and fluorine gas at their respective flow rates until 1 gram of hydrogen gas has been passed in, and 16.2 grams of fluorine gas has been passed in. Note: during the addition of the hydrogen and fluorine, monitor the temperature of the reaction chamber and do not allow its temperature to go above 500 Celsius—turning off the meeker burners or whatever heat source you used temporarily may need to be done in order to keep the reaction below 505 Celsius, as the electric arc in the system will contribute some heat of its own. Once the desired amount of hydrogen gas and fluorine gas has been added, turn off the meeker burners/heat source, then at the same time turn off power supply, and then stop the addition of argon gas. Now, detach the power supply clamps, then detach the hoses leading to the gas supplies (argon, hydrogen, and fluorine), and then detach the hoses leading to the condenser, and then using any desired means, begin to rate the entire apparatus at 150 RPM until the temperature inside the reaction chamber cools to room temperature. Once the temperature of the reaction chamber has cooled to room temperature, stop rotating the apparatus, and then disassemble the apparatus, and do the following: while dissembling the apparatus, keep the lower adapter containing the platinum cathode in place, and keep the adapters where the gases are introduced in place, so that no liquid or fluid escapes as the apparatus is disassembled. Remove the upper adapter containing the platinum anode, and then scrape off the residue that will be stuck to it, and place this residue into a clean beaker. Thereafter, simply take the partially disassembled apparatus, and pour out the fluid into the same beaker just used. Now, scrape off any residue stuck to the platinum cathode, and place this residue into the same beaker as just used. Note: now the rest of this apparatus can be dissembled for cleaning. Now, to the contents contained in the beaker, pour in 1500 milliliters of ice water, and then stir this new mixture for about 30 minutes or until the ice melts. Note: a two-phase mixture will result—never mind this as the desired product is insoluble in both layers, and will sink to the bottom of the beaker. After stirring the mixture for about 30 minutes, filter-off the insoluble solids, using a Buchner funnel under suction filtration, and then wash this filtered off insoluble material with three 250-milliliter portions of dry fresh diethyl ether, followed by three 1500-milliliter portions of hot water, and then suction dry the washed filtered-off product as best as possible. Finally, remove the washed suction dried product from the Buchner funnel, and place it into a clean crucible, and then place this crucible into an oven and bake at 130 Celsius for 6 hours. After 6 hours, the desired product will be completely dry and ready for use—after turning off the oven, and allowing the desired product to cool to room temperature, remove it from the oven, and store it in a glass jar until use.



All nickel apparatus for generating 7,12-dimethyl-4,10-dioxa-3,5,9,11-tetrachromiumtricyclo [6.3.1.0(2,6)]dodeca-1,6,8(12)-triene 3,3,5,5,9,9,11,11,-octaoxide.

Theoretical Preparation 60: Xenon trioxide; Xenic trioxide; Trioxide of Xenon; Xenic acid anhydride; LYP-109

	
Expanded structure	3D structure

Molecular data:

Molecular Formula = O_3Xe
Formula Weight = 179.2944916
Composition = O(26.77%) Xe(73.23%)

Molar Refractivity = Not available
Molar Volume = 372 ± 9.0 cm^3
Parachor = 892.0 ± 9.275.0 cm^3
Index of Refraction = 3.0012 ± 0.029
Surface Tension = Not available
Density = 4.45 ± 0.0490 g/cm^3
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 179.89219 Da
Nominal Mass = 180 Da
Average Mass = 179.2945 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Proven Flammability:	Proven Sensitivity:	Reactivity:	Partial Physical aspect:
8096; powerful explosive! (Estimated from German et. Al. vol. 1, 1997)	Non-flammable; detonates when touched by a spark or flame, or upon strong concussion. Forms super powerful explosives when mixed with reducing agents and touched by a spark, flame, or electric arc.	Stable towards non-flame heat, but will detonate when strongly and rapidly heated (a sample in a beaker will detonate if heated with a Bunsen burner); will detonate when touched by a spark, flame, or electric arc; non-sensitive to pressure or friction, but will detonate if struck by a hammer. A primary explosive.	Super strong oxidizer! May explode in contact with organic material; stable towards the halogens, does not react with fluorine, but is easily hydrolyzed by water forming the so called “xenic acid”, keep submerged under kerosene to protect it from moisture—may ignite spontaneously when exposed to combustible material in the presence of moisture or water. May ignite spontaneously when mixed with flammable liquids such as ethers, ketones, aldehydes, carboxylic acids, and alcohols, but can be safely stored for prolonged periods of time under low volatile hydrocarbon liquids, i.e., kerosene.	Colorless, transparent, unknown crystalline solid; Melting point estimated to range from 56 to 78 Celsius (use caution when heating as explosion may result); May sublime when gently heated under super high vacuum. Insoluble in all known solvents. Keep in a dessicator sealed airtight over sodium hydride, or keep submerged under kerosene for prolonged storage.

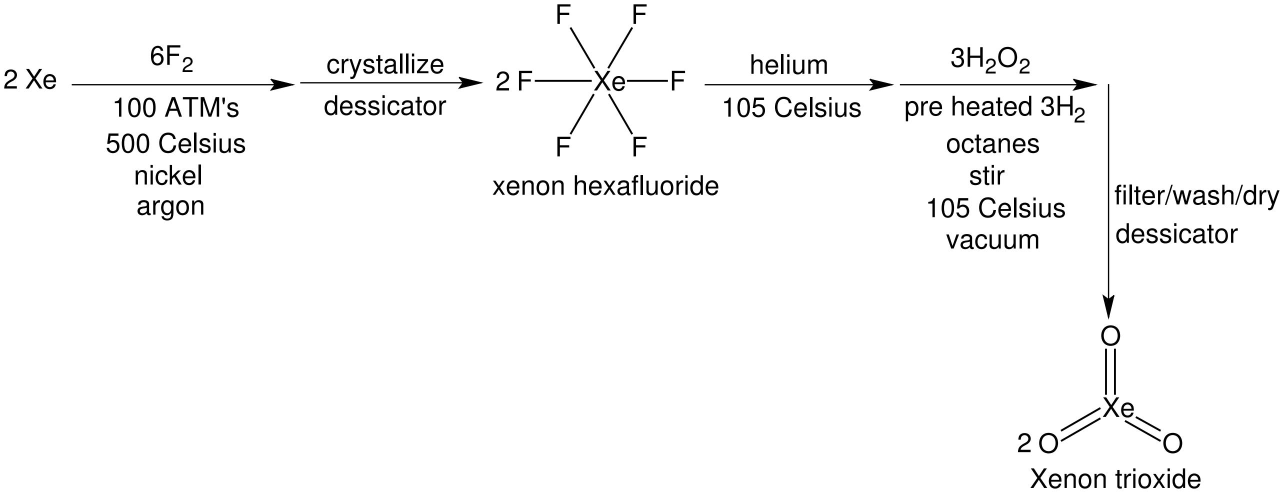
Partial Compound summary: **Note:** Xenon trioxide is a known and tested compound and has been successfully synthesized—it is being included in this book due to the rarity of it’s existence, and little information is known about this substance as it compares to practical uses in the modern explosives world. Xenon trioxide is an absolutely amazing primary explosive made from an other wise “inert gas” (according to the periodic table), and is one of only a few compounds made from an “inert gas”. Xenon trioxide forms highly explosive mixtures with reducing agents and any combustible material when touched by a spark or flame; hence, it can be used to make a variety of high explosive mixtures mainly with powdered metals, hypophosphites, hydrides, sulfur, charcoal, sulfides, sulfites, phosphites, finely ground polymers such as finely divided PVC, powdered carbohydrates such as powdered sugar, and wood dust or saw dust—however, these mixtures are untested and considered extremely dangerous to make and handle as they all easily detonate when touched by a spark, flame, or electric arc, and much more research needs to be conducted in this field. Other then that, the only current use for xenon trioxide that has actually been "partially" tested is in the use of making blasting caps and detonators in place of lead styphnate, lead azide, RDX, and other more commonly used chemicals used in current blasting caps or detonators; however currently, no military or nation on earth uses xenon trioxide as a primary explosive in blasting caps or detonators (circa 2010)—nonetheless, theoretical research has shown that xenon trioxide could make a useful priming composition in non-electric and electric blasting caps when diluted with powdered silicon dioxide (do not cure with epoxy). Also, xenon trioxide when in small granular form, could be used in the tips of explosive shells and rockets, whereby the xenon trioxide would detonate upon impact of the shell or rocket and could thereby be used to initiate explosive trains as found in blasting caps and detonators for detonating secondary explosives in the case of "point-initiating-point-detonating" fuses.

Safety Info: Use extreme caution when handling fluorine gas, which is highly toxic and very reactive; fluorine gas will ignite in the contact with many combustible materials including organic liquids and solids—use maximum ventilation. Extinguish all flames before using octanes, which are highly flammable. Keep 99% hydrogen peroxide away from oxidizable materials as fire may result. Hydrogen gas forms explosive mixtures with air so use maximum ventilation and avoid contact with all sources of ignition. Use maximum ventilation and avoid inhalation of the fumes when handling diethyl ether, which is a narcotic in high concentrations and forms explosive mixtures with air, use caution. Also, never use ether that has been sitting on the shelf for prolonged periods of time as explosive peroxides may have formed—always perform the peroxide test before using ether that has been in prolonged storage.

Materials:

1. 97 grams of xenon gas	5. 28 grams of 99% hydrogen peroxide
2. 84.2 grams of fluorine gas	6. 1.66 grams of hydrogen gas
3. Argon gas from tank	7. Helium gas from tank
4. 500 milliliters of octanes	8. 1500 milliliters of fresh diethyl ether

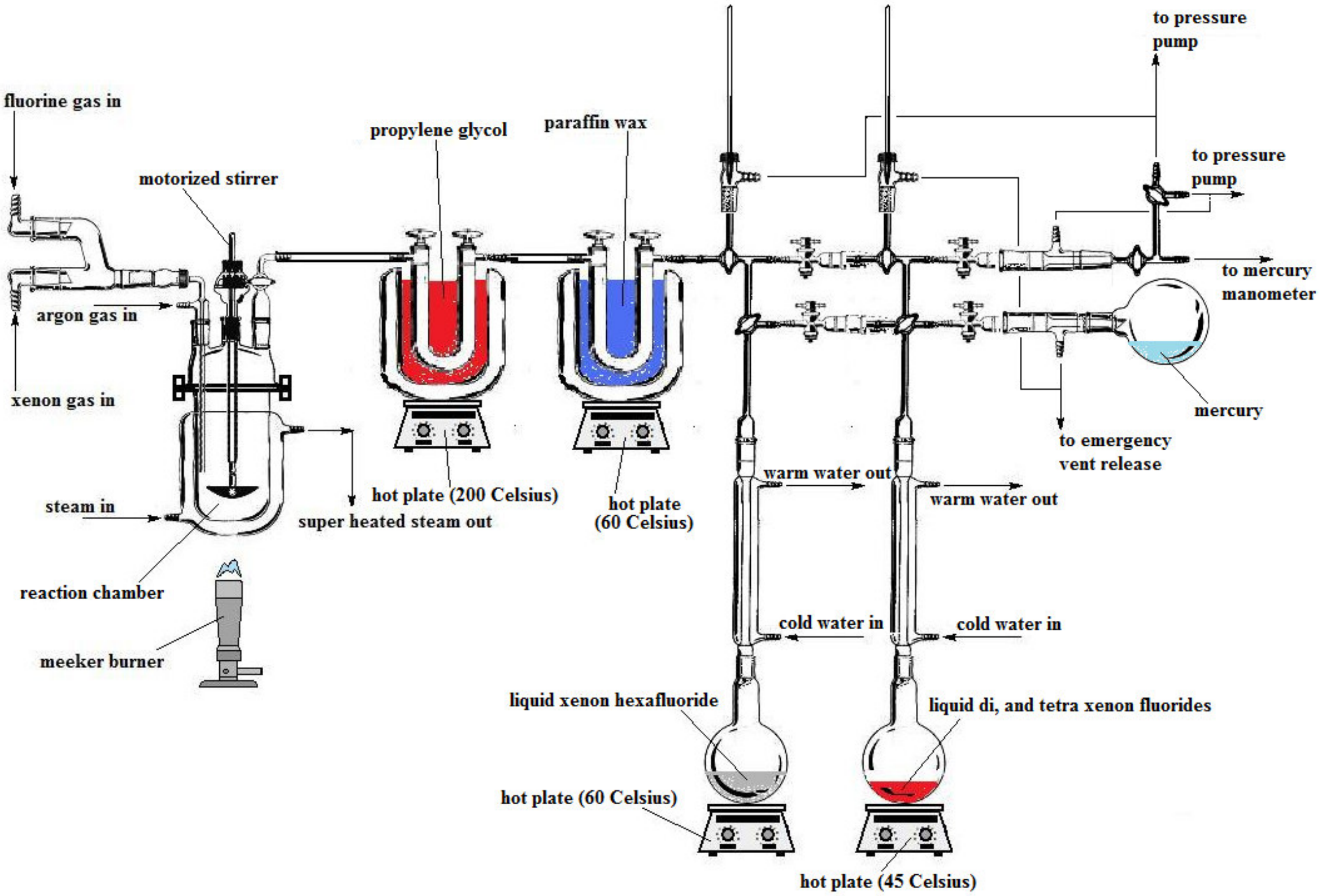
Reaction Equation:



Preparation:

Step 1: Preparation of xenon hexafluoride

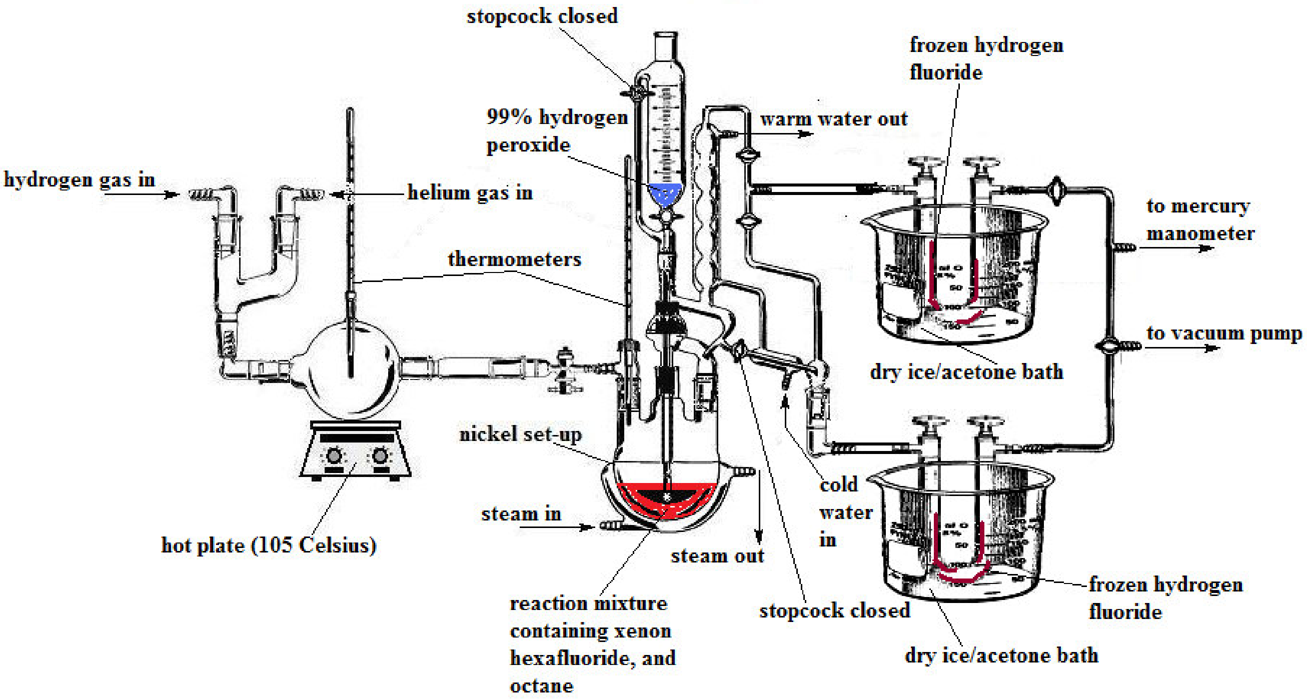
Into an exhaustive and advanced apparatus as illustrated below, begin a argon gas purge at a flow rate of 500 mg per/minute, and turn on the pressure pumps, and bring the pressure inside the reaction chamber to 100 ATM's. Thereafter, turn on the meeker burner, and bring the temperature of the reaction chamber to 500 Celsius. Then turn on the hot plates as illustrated and indicated to their respective temperatures. When the pressure inside the reaction temperature reaches 100 ATM's, and a temperature of 500 Celsius, and the hot plates reach their respective temperatures, begin to pass into the reaction chamber xenon gas at a flow rate of 0.023 mg per/minute, and at the same time begin to pass into the reaction chamber fluorine gas at a flow rate of 0.020 mg per/minute. Continue to pressurize the reaction chamber at 500 ATM's at a temperature of 500 Celsius until a total of 97 grams of xenon gas has been added, and a total of 84.2 grams of fluorine gas has been added. Once all the xenon and fluorine gases have been added, turn off the xenon gas source, and turn off the fluorine gas source, but continue to purge the apparatus with argon, and turn off the meeker burner, and allow the temperature inside the reaction chamber to cool to room temperature. Note: during this cool down period continue to purge and pressurize the apparatus at 100 ATM's for 5 hours. After 5 hours, turn off the pressure pumps, and then release the emergency relief valve to bring the pressure inside the apparatus to STP, then turn off the argon gas source. Now, as according to the illustration of the apparatus, the hot plate heating the liquid xenon hexafluoride should be turned off, and then the flask containing the liquefied xenon hexafluoride should be removed from the apparatus, and the liquid xenon hexafluoride then poured into a crystallizing dish, and then immediately stored in a dessicator over sodium hydride and allowed to cool and crystallize forming beautiful colorless crystals of xenon hexafluoride. Once the xenon hexafluoride has cooled and crystallized, keep stored airtight in the same dessicator until use in step 2.



All nickel apparatus for the preparation of xenon hexafluoride.

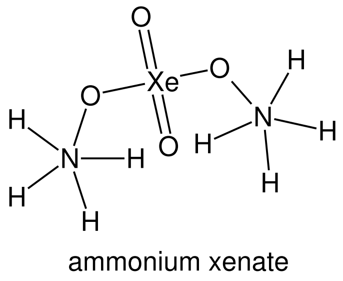
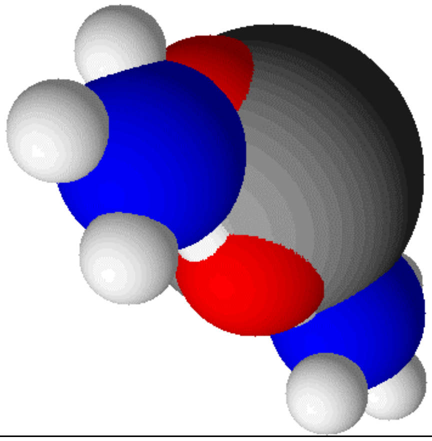
Step 2: preparation of xenon trioxide

Into yet another highly advanced apparatus as illustrated below, turn on the hot plate as illustrated to the left, and heat to 105 Celsius. Thereafter, into the reaction flask as illustrated, place 500 milliliters of octanes (a mixture of octane isomers obtained from gasoline), followed by 135 grams of xenon hexafluoride as prepared in step 1, and then begin to pass steam through the outer jacket of the reaction flask as illustrated. Now, when the hot plate reaches 105 Celsius, and the temperature inside the reaction flask reaches 105+ Celsius, turn on the vacuum pump to 400mm of mercury, and then immediately begin to pass in helium gas into the apparatus at a flow rate of 500 mg per/minute, followed by hydrogen gas at a flow rate of 0.059 mg per /minute. Immediately thereafter, pour into the addition funnel as illustrated 28 grams of 99% hydrogen peroxide, and then begin a very slow drip of the hydrogen peroxide into the reaction mixture. Then turn on the motorized stirrer, and rapidly stir the reaction mixture throughout the addition process. Continue to heat the corresponding parts of the apparatus as illustrated, and continue to pass in helium gas and hydrogen gas, until 1.66 grams of hydrogen gas has been added. Note: the addition of the hydrogen gas needs to be slow in along with a very slow addition of the 99% hydrogen peroxide so as both components are added equally at the same time. Now, once all the desired amount of hydrogen gas and hydrogen peroxide has been added, turn off the hydrogen gas source, but continue to purge the apparatus with helium gas, and maintain a vacuum of 400mm of mercury for 2 hours with rapid stirring. Note: during the reaction process, the octanes will reflux and condense in the bulb condenser as illustrated, and will return back to the reaction mixture. After 2 hours, turn off the hot plate, and turn off the steam source, and turn off the vacuum pump, and allow the reaction mixture to cool to room temperature, and allow the pressure inside the apparatus to come to STP. Note: during the reaction hydrogen fluoride gas will be steadily evolved and will condense in the dry ice/acetone baths forming colorless crystals. This hydrogen fluoride can be discarded if desired, or it can be added to water to form hydrofluoric acid for some future process—note: store the hydrofluoric acid in a Teflon bottle, as it corrodes glass. In any sense, once the reaction mixture reaches room temperature, carefully disassemble the apparatus, and then filter-off the insoluble desired product from the reaction mixture using a Buchner funnel under suction. Thereafter, wash the filtered-off desired product of xenon trioxide with three 500-milliliter portions of fresh diethyl ether, and then suction dry as best as possible. Finely, remove the filtered washed desired product from the Buchner funnel and store the desired product in a dessicator over phosphorus pentoxide until use.



All nickel apparatus for preparation of xenon trioxide.

Theoretical Preparation 61: Ammonium xenate; Xenic acid ammonium salt; diamonium xenate; DAX;

	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $(NH_4)_2XeO_4$
Formula Weight = 231.3708
Composition = H(3.48%) N(12.1%) O(27.65%) Xe(56.77%)

Molar Refractivity = Not available
Molar Volume = 299 ± 6.70.0 cm^3
Parachor = 1,090.0 ± 12.0668 cm^3
Index of Refraction = 2.998 ± 0.1073
Surface Tension = Not available
Density = 2.98 ± 0.2609 g/cm^3
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 198.999 Da
Nominal Mass = 209.4598 Da
Average Mass = 210.00988 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6802; estimated using infrared spectra	Non-flammable; does not burn even when strongly heated and ignited. Forms explosive mixtures with reducing agents.	Remarkably stable. Stable towards moderate heat, percussion, concussion, pressure, and friction. A secondary explosive. Requires a blasting cap or detonator for initiation.	Powerful oxidizing agent! Reacts violently with fluorine, but is non-reactive to the other halogens. Stable towards other strong oxidizers including calcium hypochlorite, potassium permanganate, and potassium dichromate, even when heated. Keep out of contact with combustible materials especially flammable liquids—may ignite causing fire; however, is stable and non-reactive to hydrocarbons, and chlorinated or brominated hydrocarbons.	Colorless crystals, powder or granules; crystal structure very similar to that of ammonium nitrate. Melting point: decomposes into xenon, nitrous oxide, and water when heated to above 230 Celsius. Very soluble in water and alcohol ~up to 40% by weigh solutions in water and alcohol; insoluble in ether, methylene chloride, hydrocarbon solvents, and most other solvents. Slightly soluble in liquid ammonia.

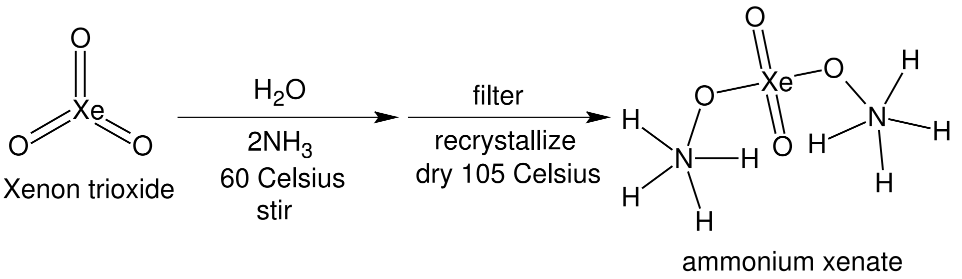
Estimated Compound summary: Ammonium xenate is a fascinating xenon containing addition compound containing ammonium ion groups with interesting properties very similar to ammonium nitrate and ammonium perchlorate. Ammonium xenate has a remarkably and unexpected high detonating velocity making it useful for making explosive munitions, but it has to be dead pressed into shells under high pressure, and initiated with a standard blasting cap or detonator. Like ammonium nitrate, ammonium xenate forms prills when mixed with 5% fuel oil, and primed with a standard blasting cap or detonator. These prills form safe high explosives that are very stable and can be used in general blasting operations for military and commercial use. Ammonium xenate demonstrates very usefulness in making high performance solid rocket fuels when mixed with powdered metals, mainly powdered aluminum or magnesium, and cured with a suitable epoxy agent. Ammonium xenate can also be used to make high performance gun propellants, when slurried with nitroglycerine or any other poly nitrated alcohols, mixed with nitrocellulose, and then cured with a high energy plasticizer. These high performance gun propellants are very powerful and can produce chamber pressures of up to 120,000 psi; however, these chamber pressures are way to high for small arms weapons, and is more useful in making propellants for mortar shells, artillery shells, ship deck guns, ect. Ammonium xenate can also be used to make a variety of pyrotechnic mixtures for various operations, but due to the high cost of producing ammonium xenate, its use in making pyrotechnic mixtures is surpassed by its more effective and usefulness in making high performance solid rocket fuels and propellants, the latter primarily in making propellants to launch mortar rounds—due to its high gas ration upon ignition, and it's amazing pressures produced upon ignition of these ammonium xenate propellants, ammonium xenate propellants used in mortar shells can launch heavy mortar shells up to extremely high distances, 4 times that of ordinary mortar propellants currently used by modern militaries.

Safety Info: Use caution when handling xenon trioxide, as it is a primary explosive—use the appropriate precautions. 20% aqueous ammonia is highly irritating to the nose and throat, use proper ventilation.

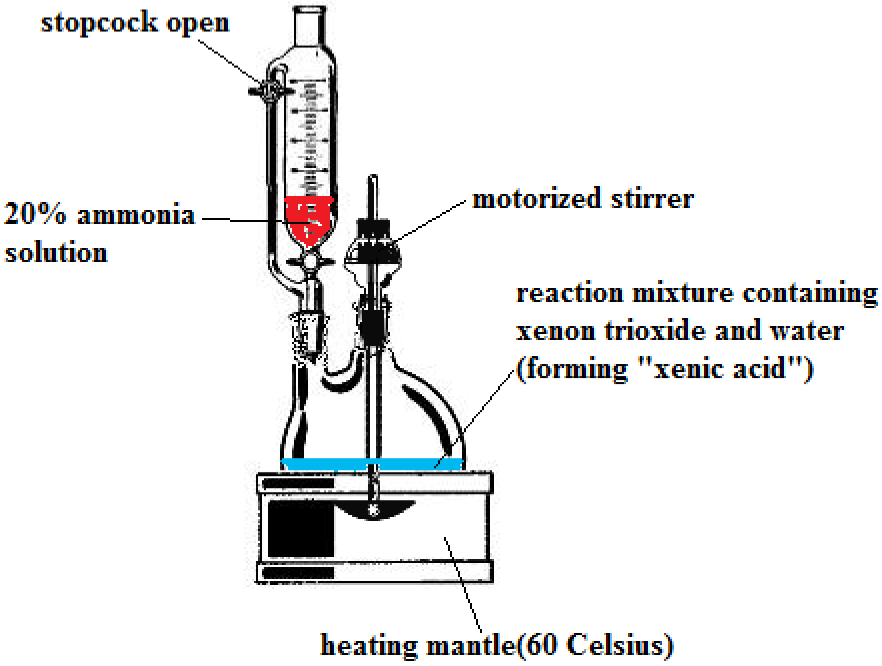
Materials:

1. 500 milliliters of distilled water	3. 100 grams of a 20% aqueous ammonia solution
2. 102 grams of xenon trioxide (prepared in theoretical preparation 60, step 2)	

Reaction Equation:

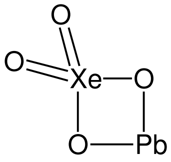
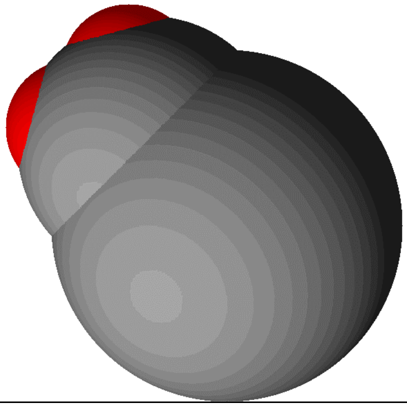


Preparation: Into an apparatus as the one illustrated below, pour in 500 milliliters of distilled water, and then slowly add in, in small portions at a time, 102 grams of xenon trioxide over a period of about 30 minutes, with constant stirring. Note: during the addition of the xenon trioxide to water, it will hydrolyze forming the so-called xenic acid (H_2XeO_4). After the addition of the xenon trioxide to the water in the reaction flask, continue to stir the reaction mixture containing the now xenic acid for about 1 hour at room temperature. After 1 hour, attach an addition funnel as illustrated, and then pour in 100 grams of a 20% aqueous ammonia solution into the addition funnel. Thereafter, turn on the heating mantle, and heat the reaction mixture containing the xenic acid to 60 Celsius, and when this reaction mixture reaches 60 Celsius, begin to slowly drip into the reaction mixture the 20% aqueous ammonia solution from the addition funnel over a period of 45 minutes, while heating the reaction mixture at 60 Celsius with constant stirring. After the addition of the 20% aqueous ammonia to the reaction mixture, continue to stir the reaction mixture, but turn off the heating mantle and allow the reaction mixture to cool to room temperature. Continue to stir the reaction mixture during this cool down period. Once the reaction mixture has cooled to room temperature, stop the stirring, and then disassemble the apparatus, and then quickly filter the reaction mixture using suction filtration, and then pour the filtered reaction mixture into a suitable sized beaker, and then heat this reaction mixture to 105 Celsius to boil off the water, and concentrate the ammonium xenate dissolved therein, i.e. perform a recrystallization process, collecting the ammonium xenate crystals on the same filter after each crop. Once the bulk of the ammonium xenate has been recrystallized and recovered on the same filter, suction dry as best as possible. Finely, place this suction dried ammonium xenate crystals onto a crystallizing dish, and place this dish into an oven, and heat to 105 Celsius for 4 hours to completely dry the ammonium xenate. After 4 hours, turn off the oven, and allow the ammonium xenate to cool to room temperature, once it has, store the ammonium xenate in a plastic or glass container sealed air-tight, and store in a cool dry place until use.


stopcock open
20% ammonia solution
motorized stirrer
reaction mixture containing xenon trioxide and water (forming "xenic acid")
heating mantle(60 Celsius)

Simple setup for preparing ammonium xenate. Note: before heating and adding the ammonia solution, carefully add in small portions of the xenon trioxide to the water in the reaction flask so as to form xenic acid.

Theoretical Preparation 62: Lead-II-xenate; Xenic acid lead salt; L2X; Plumbous xenate

 lead-II-xenate	
Expanded structure	3D structure

Molecular data:
Molecular Formula = PbO_4Xe
Formula Weight = 402.4906
Composition = Pb(51.69%) O(15.83%) Xe(32.48%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = 1,996.0 ± 21.0932 cm^3
Index of Refraction = 6.0091 ± 0.2598
Surface Tension = Not available
Density = 11.00879 ± 0.0916 g/cm^3
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 404.866956 Da

Nominal Mass = 405 Da
Average Mass = 404.2737 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
5590	Non-flammable; explodes when heated or ignited. Does not form flammable or explosive mixtures with combustible materials except powered metals, sulfur, and charcoal forming shock sensitive and highly explosive mixtures.	Stable under normal conditions, but is shock sensitive, and sensitive to percussion, concussion, heat, fire, electric arc, pressure, and friction. Samples will explode if struck by a hammer. Sensitivity similar to lead azide, and samples should be diluted with glucose for stability. A primary explosive	Generally non-reactive, but reacts violently with fluorine and chlorine, but stable towards bromine and iodine. Samples are stable towards strong oxidizers such as potassium permanganate, potassium dichromate, and calcium hypochlorite. Does not react with organic materials such as alcohols, ethers, ketones, aldehydes, and carboxylic acids. Stable towards strong acids such as concentrated hydrochloric or sulfuric acid. Should be stored under kerosene for prolonged periods of time, or diluted with glucose for use in blasting caps and detonators.	Bright red brilliant amorphous powder. Melting point: explodes when heated to above 90 Celsius. Insoluble in water and all known solvents.

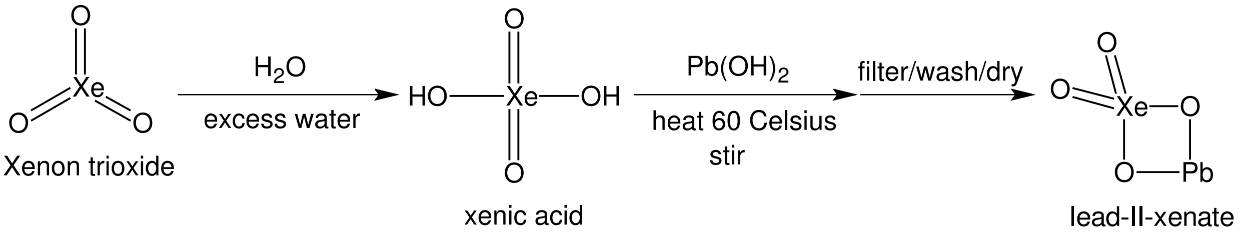
Estimated Compound summary: Lead-II-xenate is another xenon salt of xenic acid. It is a primary explosive with properties similar to lead azide, but it is a tad bit more sensitive then lead azide. Even though lead azide is cheaper and the most widely used initiating explosive used widely in blasting caps and detonators, lead xenate portrays a higher sensitivity and hence can be used as a priming explosive in non-electric and electric blasting caps as it is easily detonated when touched by a spark or flame or heat, and it can be used to make shortened blasting caps and detonators where an initiating agent like lead azide is commonly used is not necessary. These blasting caps and detonators can be made by dead pressing RDX into the bottom off the blasting cap or detonator, and then lead xenate diluted with 20% glucose then gently pressed into the blasting cap or detonator, and this is all that would be needed, as when the lead xenate is detonated by spark, flame, or heat, its energy upon detonation is sufficient enough to detonate the RDX. Lead xenate used in blasting caps or detonators do not need the more common lead styphnate and barium chromate priming compositions, and hence lead xenate blasting caps and detonators would only have to be manufactured using the two explosives RDX and lead xenate, saving time and space. In other uses, lead xenate can be used in point-initiating-point-detonating fuses for mortar shells and other low velocity projectiles such as 40mm grenades, but lead xenate should not be used in high velocity shells due to its sensitivity even when diluted with 20% glucose, as the shock from firing these high velocity projectiles could cause the lead xenate to detonate prematurely.

Safety Info: Use caution when handling xenon trioxide, as it is a primary explosive—use the appropriate precautions. Wear gloves when handling lead compounds and avoid inhalation of the dust and avoid skin contact.

Materials:

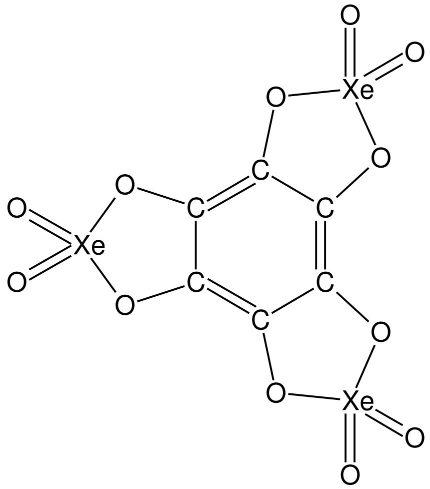
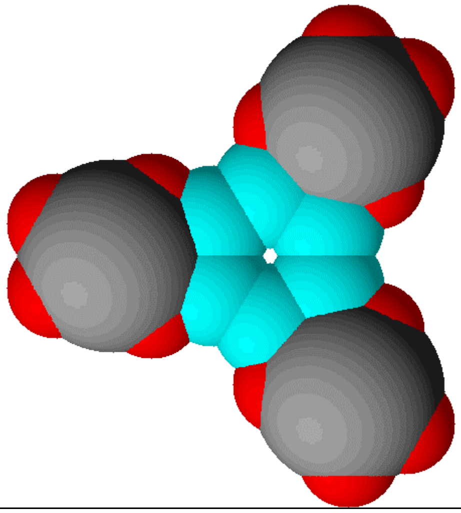
1. 102 grams of xenon trioxide (prepared in theoretical preparation 60, step 2)	3. 137 grams of freshly precipitated lead-II-hydroxide
2. 750 milliliters of distilled water	

Reaction Equation:



Preparation: Into a suitable sized beaker fitted with motorized stirrer, and sitting on top of a hot plate, place 750 milliliters of distilled water, and then slowly and carefully add in, in small portions at a time, 102 grams of xenon trioxide over a period of about 45 minutes while vigorously stirring the reaction mixture and maintaining its temperature around 20 Celsius. Note: during the addition of the xenon trioxide, xenic acid will form in solution. After the addition of the xenon trioxide, continue to stir this acidic reaction mixture for about 15 minutes at room temperature, and then turn on the hot plate, and bring the temperature of the reaction mixture to 60 Celsius with constant stirring. Once the acidic reaction mixture containing the xenic acid reaches a temperature of around 60 Celsius (do not exceed 60 Celsius), begin to add in, in small portions at a time, 137 grams of freshly precipitated lead-II-hydroxide over a period of about 1 hour while heating the reaction mixture to 60 Celsius with vigorous stirring. Note: monitor the temperature carefully, and do not allow it to rise above 80 Celsius. After the addition of the lead-II-hydroxide, continue to heat the reaction mixture at 60 Celsius under rapid stirring for 4 hours. After 4 hours, turn off the hot plate, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, remove the motorized stirrer, and then filter-off the insoluble desired product of lead xenate using a Buchner funnel under suction. Then simply wash this filtered-off desired product with three 500-milliliter portions of warm water, and then suction dry as best as possible. Finally, remove the suction dried product from the Buchner funnel and place it onto a shallow pan, and allow it to air dry for 4 days. After 4 days, the desired product of lead xenate will be completely dry and should then be stored in amber glass bottles in a cool dry place until use.

Theoretical Preparation 63: [1,3,2]dioxaxenate[4,5-*e*][1,3,2]benzodioxaxenate[3,5-*e*][1,3,2]benzodioxaxenate[1,5- *e*] 2,2,5,5,7,7-hexaoxide; Benzene tri-xenate; DBBH; DBBH-tri-xenate; 1,2,3,4,5,6-trixenate benzene

	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6O_{12}Xe_3$
Formula Weight = 657.9458748
Composition = C(10.95%) O(29.18%) Xe(59.87%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 659.661315 Da
Nominal Mass = 660 Da
Average Mass = 657.9459 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
7824	Non-flammable; does not form explosive mixtures with reducing agents.	Very stable explosive. Non-sensitive to shock, percussion, concussion, heat, pressure or friction. Requires significant blasting cap or detonator for proper initiation, A secondary explosive.	Generally non reactive, but reacts violently with fluorine, but very stable and non-reactive to the other halogens. Does not react with oxidizers, or reducing agents even when heated.	White crystalline powder. Melting point estimated to be around 190 to 210 Celsius; decomposes when heated to above 450 Celsius. Violent decomposition results when strongly and rapidly heated to above 500 Celsius. Insoluble in water and all known solvents.

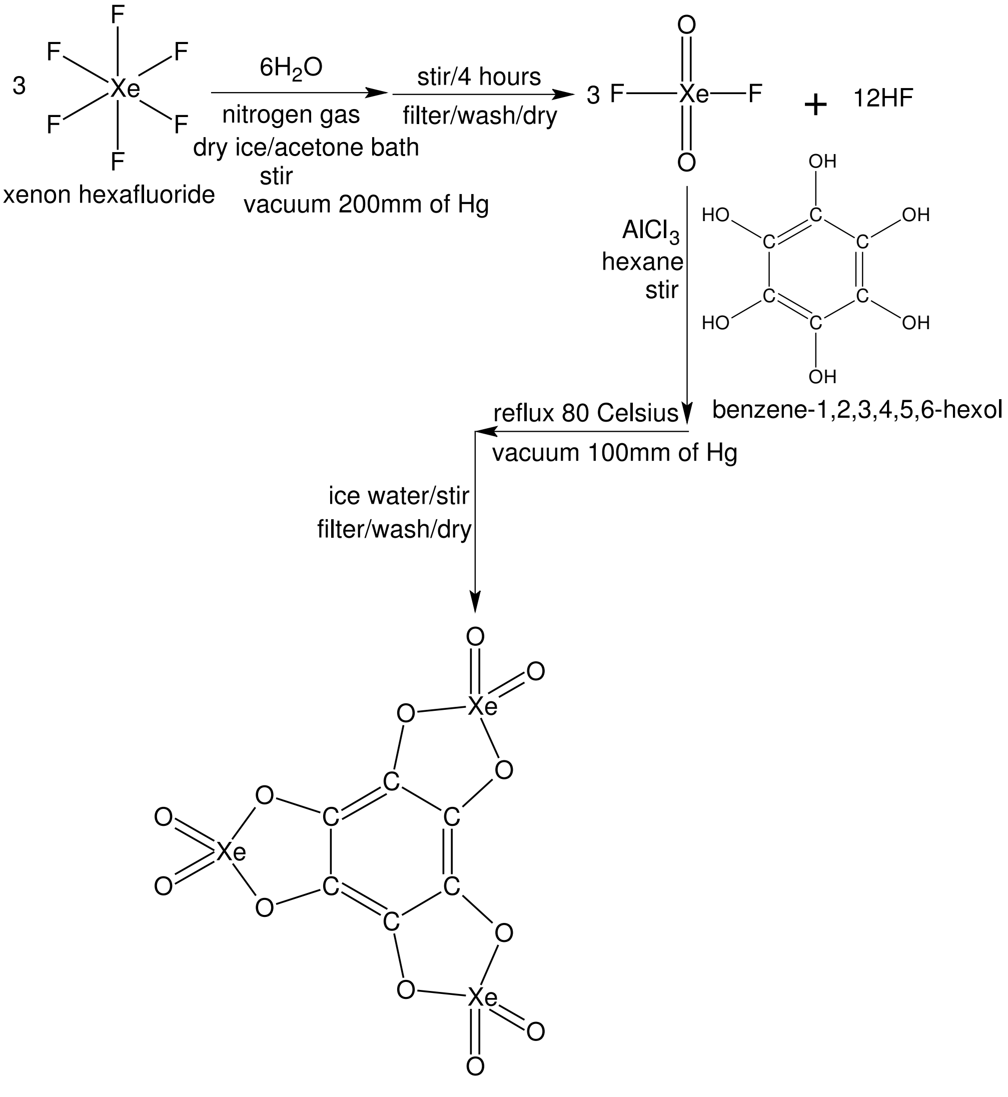
Estimated Compound summary: [1,3,2]dioxaxenate[4,5-*e*][1,3,2]benzodioxaxenate[3,5- *e*][1,3,2]benzodioxaxenate[1,5-*e*] 2,2,5,5,7,7-hexaoxide is a very stable cyclic xenon high explosive that is very stable and requires a large blasting cap or detonator for proper initiation. Due to its stability, it can easily be melted down and poured into molds for making a wide variety of explosive munitions such as mortar shells, artillery shells, shape charges, missile warheads, rocket warheads, land mines, and many other explosive munitions. [1,3,2]dioxaxenate[4,5-*e*][1,3,2]benzodioxaxenate[3,5-*e*][1,3,2]benzodioxaxenate[1,5- *e*] 2,2,5,5,7,7-hexaoxide does not burn or form deflagrating mixtures when mixed with powdered metals, and other reducing agents, and hence cannot be used to make gun propellants, rocket fuels, or other pyrotechnic mixtures.

Safety Info: Extinguish flames and sources of ignition before handling n-pentane, which is highly volatile and flammable. Use caution when handling ether, and extinguish all flames before using ether, which is highly flammable, and forms explosive mixtures with air. Avoid inhalation of ether vapors, as ether is a narcotic in high concentrations. Also, never use ether that has been in storage for prolonged periods of time without performing the peroxide test. Hexane is highly volatile and flammable, extinguish all flames before using. Wear gloves when handling anhydrous aluminum chloride, and avoid contact with water—anhydrous aluminum chloride reacts violently with water.

Materials:

1. 500 milliliters of n-pentane	5. 750 milliliters of dry hexane
2. 106 grams of xenon hexafluoride (prepared in theoretical preparation 60, step 1)	6. 100 grams of anhydrous aluminum chloride
3. 15.57 milliliters of distilled water	7. 25 grams of benzene-1,2,3,4,5,6-hexol
4. 1200 milliliters of diethyl ether	

Reaction Equation:

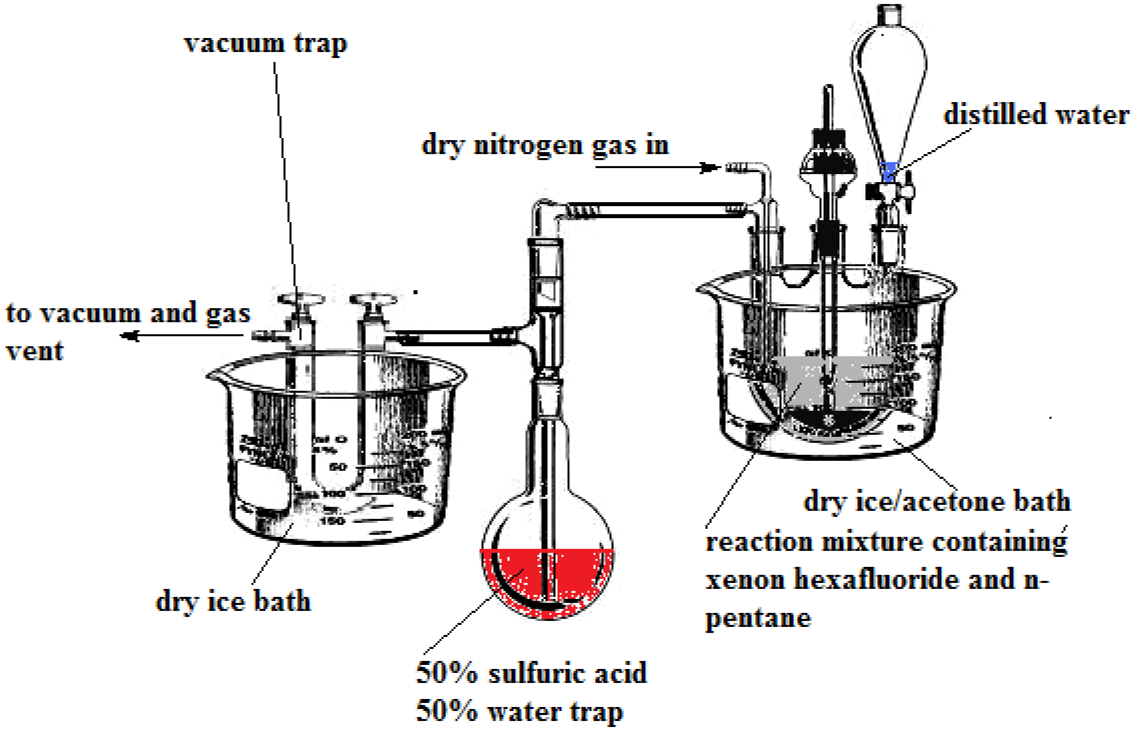


[1,3,2]dioxaxenate[4,5-*e*][1,3,2]benzodiozaxenate[3,5-e][1,3,2]benzodioxaxenate[1,5-e] 2,2,5,5,7,7-hexaoxide

Preparation:

Step 1: preparation of difluoro xenon dioxide

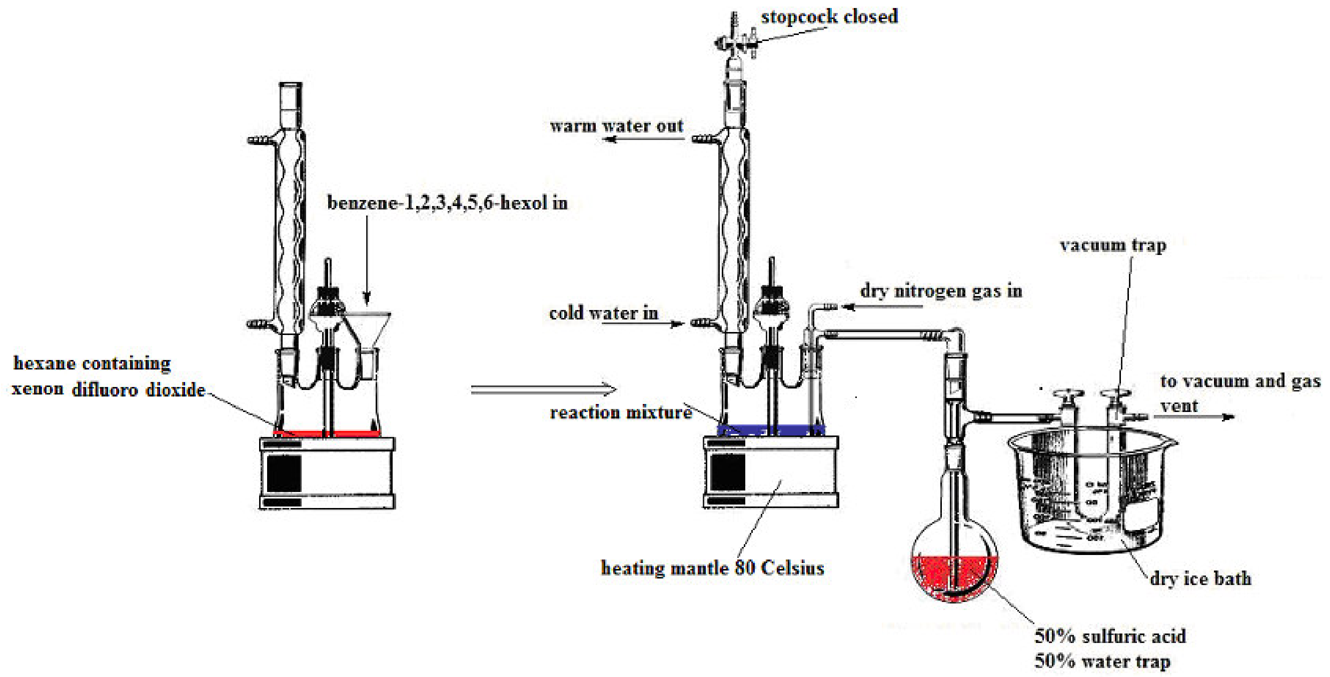
Into a setup as the illustrated below, pour in 500 milliliters of n-pentane, and then add in all at once, 106 grams of xenon hexafluoride, and then pour in 15.57 milliliters of distilled water into the separatory funnel. Thereafter, fill the outer container as illustrated with dry ice/acetone, and chill the n-pentane/xenon hexafluoride reaction mixture to chill to –100 Celsius. Then begin to purge the apparatus with dry nitrogen gas, and when the temperature of the n-pentane/xenon hexafluoride reaction mixture reaches –100 Celsius is achieved, turn on the motorized stirrer, and then turn on the vacuum pump to 200mm of mercury, and then immediately thereafter, begin to slowly drip the distilled water into the chilled reaction mixture very slowly drop by drop over a period of about 45 minutes, while rapidly stirring the reaction mixture and maintaining its temperature below –80 Celsius. Once all the distilled water has been added, close the stopcock on the addition funnel, and then continue to rapidly stir the reaction mixture at a temperature of below –80 Celsius for 4 hours under a vacuum of 200mm of mercury. Note: more acetone and/or dry ice may have to be added to the outer container due to evaporation. Note: during the reaction mixture time, and 4 hour down time, hydrogen fluoride gas will be steadily evolved and will be collected in the sulfuric acid/water trap. After stirring and cooling the reaction mixture for 4 hours, turn off the motorized stirrer, turn off the vacuum pump, and remove the reaction mixture from the dry ice/acetone bath, and allow the reaction mixture to warm to room temperature. Thereafter, filter-off the insoluble desired product using a Buchner funnel under suction, and then wash with three 150-milliliter portions of diethyl ether, and then suction dry as best as possible. Thereafter, place this suction dried desired product of difluoro xenon dioxide into a dessicator over concentrated sulfuric acid for 48 hours. After 48 hours, the difluoro xenon dioxide is ready for step 2.



All nickel setup for the preparation of difluoro xenon dioxide.

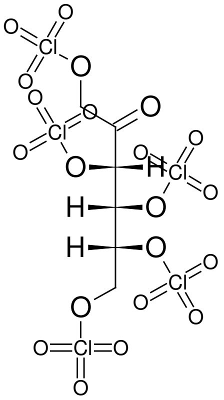
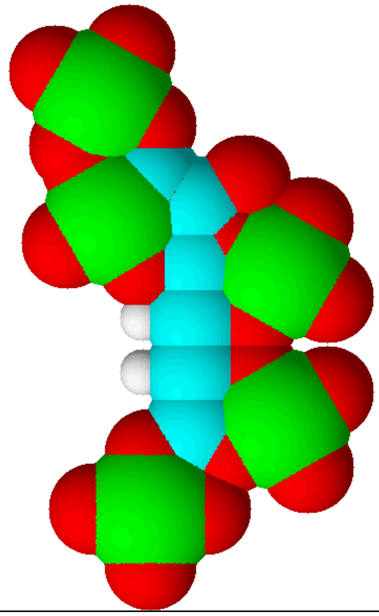
Step 2: preparation of [1,3,2]dioxaxenate[4,5-*e*][1,3,2]benzodioxaxenate[3,5- *e*][1,3,2]benzodioxaxenate[1,5- *e*] 2,2,5,5,7,7-hexaoxide

First, into the apparatus to the left of the illustration below, place 750 milliliters of dry hexane, followed by 100 grams of anhydrous aluminum chloride, and then turn on the motorized stirrer, and begin to rapidly stir the hexane reaction mixture. Immediately thereafter, add in all at once the difluoro xenon dioxide prepared in step 1, and then continue to rapidly stir this reaction mixture rapidly for 30 minutes to form a uniform dispersion. After 30 minutes, add in, in small portions at a time 25 grams of benzene-1,2,3,4,5,6-hexol over a period of 30 minutes while continuing to rapidly stir the reaction mixture. Once all the benzene-1,2,3,4,5,6-hexol has been added, assemble the apparatus to the right of the illustration, and then turn on the heating mantle to 80 Celsius with stirring. Once the reaction mixture in the right apparatus has reached 80 Celsius and a gentle reflux begins, turn on the vacuum pump to 100mm of mercury, and then continue to rapidly stir the reaction under vacuum and reflux at 80 Celsius for 6 hours. Note: during the reaction, hydrogen fluoride gas will be steadily evolved and absorbed in the sulfuric acid/water trap as like in step 1's process. After 6 hours, turn off the heating mantle and continue to rapidly stir the reaction mixture and continue the vacuum at 100mm of mercury until the reaction mixture cools to room temperature. Once the reaction mixture has cooled to room temperature, turn off the vacuum pump, and motorized stirrer, and then disassemble the apparatus, and then pour the entire reaction mixture into a large beaker filled with ice water, and then mechanically blend this new mixture on high speed for about 30 minutes. Note: when the reaction mixture is added to the ice water, a two-phase mixture will result—never mind this. Thereafter, filter-off the insoluble desired product of [1,3,2]dioxaxenate[4,5-*e*][1,3,2]benzodioxaxenate[3,5- *e*][1,3,2]benzodioxaxenate[1,5- *e*] 2,2,5,5,7,7-hexaoxide, using a Buchner funnel under suction, and then wash with three 250-milliliter portions of diethyl ether, and then suction dry as best as possible. Finally, place this suction dried desired product into an oven, and bake at 110 Celsius for 8 hours. After 8 hours, turn off the oven and allow the desired product to cool to room temperature. Once it has, remove it from the oven and store in any desired container in a cool dry place until use.


stopcock closed
warm water out
benzene-1,2,3,4,5,6-hexol in
vacuum trap
dry nitrogen gas in
cold water in
hexane containing xenon difluoro dioxide
to vacuum and gas vent
reaction mixture
heating mantle 80 Celsius
dry ice bath
50% sulfuric acid 50% water trap

All nickel apparatuses for the production of [1,3,2]dioxaxenate[4,5-e][1,3,2]benzodioxaxenate[3,5-e][1,3,2]benzodioxaxenate[1,5- e] 2,2,5,5,7,7-hexaoxide.

Theoretical Preparation 64: (2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate; Pentaperchlory fructose; 2R-3R-4S-perchloryl fructose; FRU-009

 (2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_6H_7Cl_5O_{21}$
Formula Weight = 592.37218
Composition = C(12.17%) H(1.19%) Cl(29.92%) O(56.72%)

Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 589.792246 Da
Nominal Mass = 590 Da
Average Mass = 592.3722 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
8108	Non-flammable; deflagrates when rapidly when heated to above 250 Celsius. Forms deflagrating mixtures with reducing agents especially powdered metals, sulfur, and charcoal.	Stable. Non-sensitive to shock, heat, percussion, concussion, pressure, and friction; however, a sample will detonate when strongly struck by a sledgehammer. Requires blasting cap or detonator for proper initiation. A secondary explosive.	Generally non-reactive; Reacts violently with fluorine leading to an explosion. Stable towards the other halogens, and is un-reactive to strong oxidizers even when heated. Reacts with xenon trioxide forming a complex, which is a powerful explosive with an estimated detonating velocity of over 10,000—this complex with xenon trioxide has an unknown structure!	Colorless cyndrical brilliant crystals, or white powder or granules. Melting point: 193 Celsius; Boiling point: decomposes violently when heated to above 250 Celsius. Insoluble in water and all known solvents.

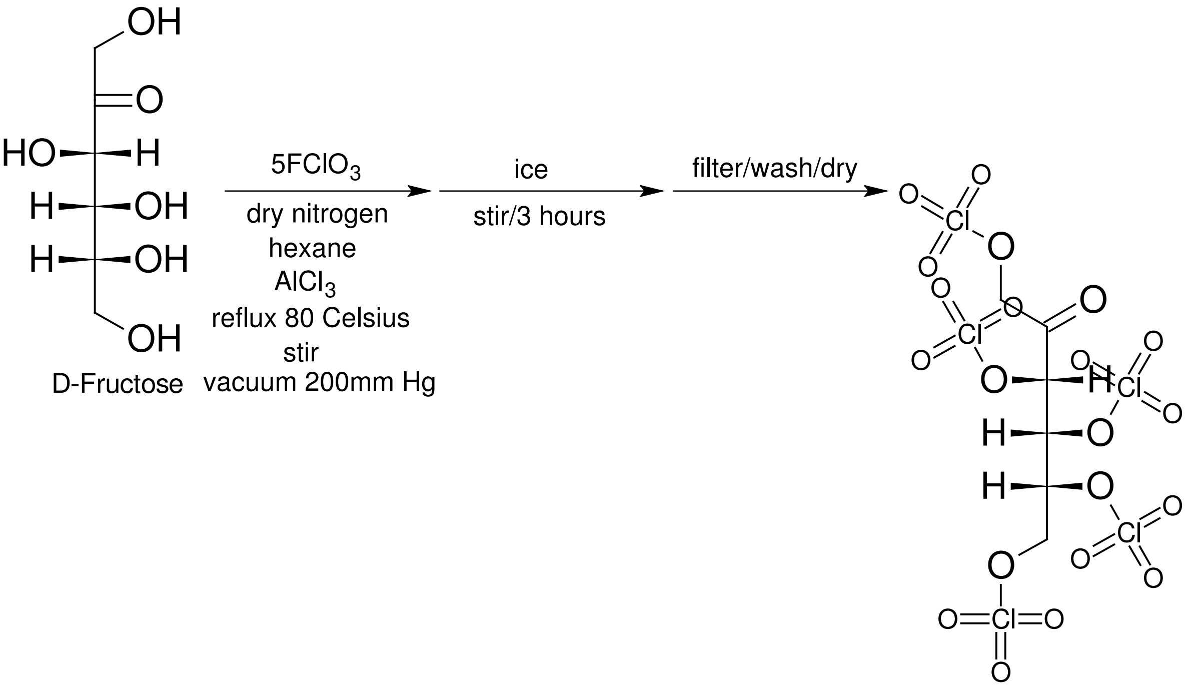
Estimated Compound summary: (2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate is an interesting perchloryl containing carbohydrate of D-fructose. The compound is remarkably stable under normal conditions and can be melted down and poured into molds for making a variety of explosive munitions including shape charges, mortar shells, artillery shells, missile warheads, and rocket warheads. (2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate forms deflagrating explosive mixtures when mixed with reducing agents such as powdered metals, sulfides, sulfur, charcoal, hypophosphites, and many others, and hence, can be used to make a variety of high performance gun propellants, and solid high performance rocket propellants, the latter when mainly mixed with powdered aluminum or magnesium and cured with epoxy. (2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate forms a interesting and amazing flexible plastic foam like explosive when mixed with polypropylene glycol and a fluorine containing high energy plasticizer and then cured in an oven. This flexible foam like explosive can then be used to form sheet explosives for demolition operations when properly primed. (2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate as previously mentioned, forms an unknown high explosive when mixed with xenon trioxide under gentle heating with stirring in an inert solvent. This xenon containing unknown complex explosive compound has been estimated to have a detonating velocity of over 10,000.

Safety Info: As usual, extinguish all flames before using hexane, which is highly volatile and flammable. Wear gloves when handling anhydrous aluminum chloride and avoid contact with moisture—anhydrous aluminum chloride reacts vigorously with water so use caution. Use extreme care, and maximum ventilation when handling perchloryl fluoride gas, which is highly reactive and can ignite on contact with combustible materials, use caution and avoid inhalation of the gas. Use caution when handling ether, and extinguish all flames before using as ether is highly flammable, and forms explosive mixtures with air. Avoid inhalation of ether vapors, as ether is a narcotic in high concentrations. Also, never use ether that has been in storage for prolonged periods of time without performing the peroxide test.

Materials:

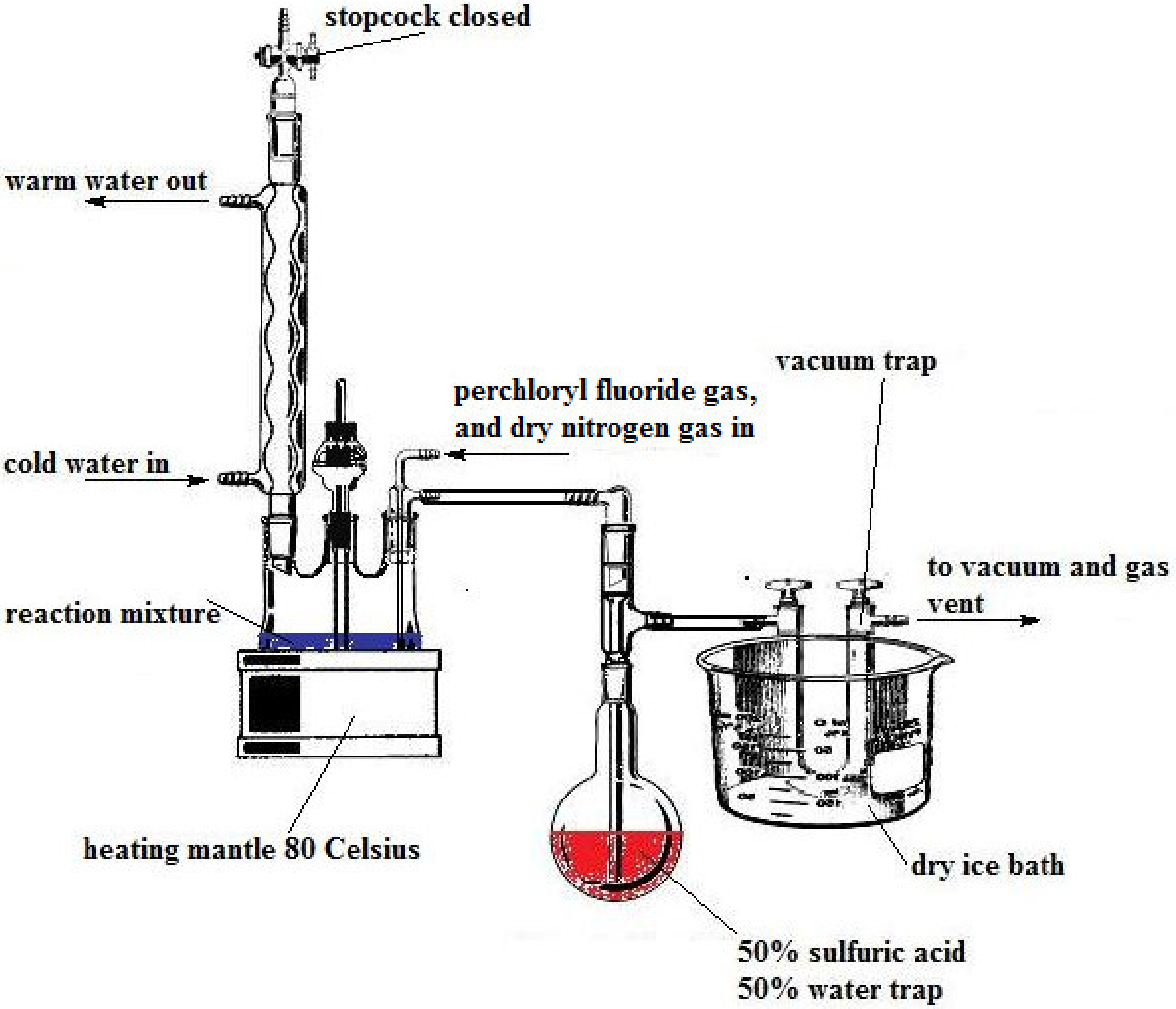
1. 750 milliliters of hexane	4. 278 grams of perchloryl fluoride gas
2. 100 grams of anhydrous aluminum chloride	5. 1500 milliliters of diethyl ether
3. Nitrogen gas from tank	6. 98 grams D-fructose

Reaction Equation:



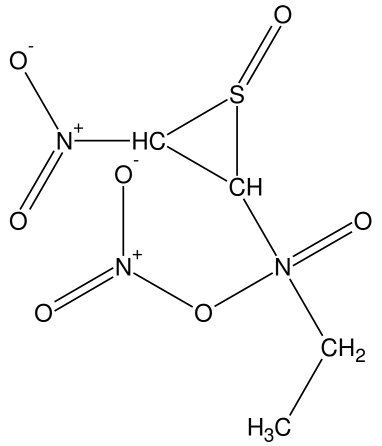
(2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate

Preparation: Into a familiar apparatus as illustrated below, pour into the three-neck flask 750 milliliters of hexane, and then add in all at once, 100 grams of anhydrous aluminum chloride, followed by 98 grams D-fructose, and then seal the apparatus, and then begin a nitrogen gas purge by pumping in dry nitrogen gas at a flow rate of 0.750 mg per/minute, and then immediately thereafter, turn on the heating mantle and begin a gentle reflux of the reaction at 80 Celsius, when the reaction mixture begins to reflux, turn on the motorized stirrer and then rapidly stir the reaction mixture for 5 minutes to form a uniform dispersion of the reactants in the reaction mixture. After 5 minutes turn on the vacuum pump to 200mm of mercury, and then immediately thereafter, begin to pass into the reaction mixture dry perchloryl fluoride at a flow rate of 0.98 mg per/minute while continuing to rapidly stir the reaction mixture and maintaining its temperature under gentle reflux at 80 Celsius. Continue to purge the apparatus with nitrogen under vacuum and continue to pass in dry perchloryl fluoride gas until a total of 278 grams of perchloryl fluoride gas has been added, while continuing to rapidly stir the reaction mixture and maintaining its temperature at 80 Celsius under gentle reflux. Once all the perchloryl fluoride has been added, continue to reflux the reaction mixture at 80 Celsius under rapid stirring for about 4 hours under vacuum of 200mm of mercury. After 4 hours, turn off the vacuum pump, and open the stopcock on top of the reflux condenser (this adapter is illustrated below and is shown as “stopcock closed”), to bring the pressure inside the apparatus back to STP, and immediately thereafter, turn off the vacuum pump. Then, turn off the nitrogen gas purge, and then turn off the heating mantle and allow the reaction mixture to cool to room temperature. Note: during the cool down period, continue to rapidly stir the reaction mixture. Once the reaction has cooled to room temperature, disassemble the apparatus, and then slowly and gently pour the entire reaction mixture into a large beaker filled with ice—note: as the ice melts a two-phase mixture will result, never mind this. Then, as the ice melts, mechanically stir this cold two-phase reaction mixture using any desired means for about 3 hours, adding more ice as the previous ice melts. After stirring for 3 hours, filter-off the insoluble desired product of (2*R*,3*R*,4*S*)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate using a Buchner funnel under strong suction, and then wash this filtered-off desired product with three 500-milliliter portions of diethyl ether, followed by washing with three 500-milliliter portions of warm water, and then suction dry as best as possible. Finally, remove the washed suction dried desired product from the Buchner funnel, and place this desired product into a oven and bake at 105 Celsius for 8 hours. After 8 hours, the desired product will be completely dried, then turn off the oven and allow this dried desired product to cool to room temperature before removing it from the oven. Once the dried desired product has cooled to room temperature remove it from the oven, and store it in any desired plastic or glass container in a cool dry place until use.



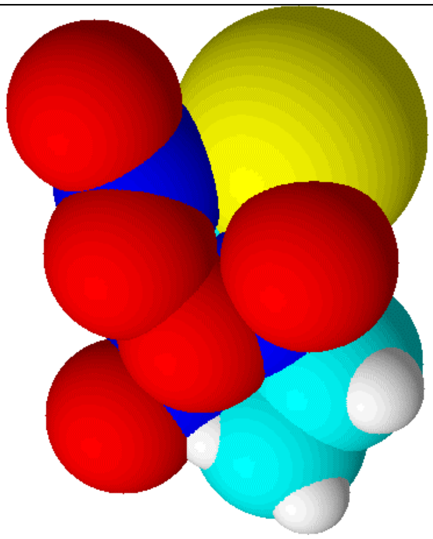
Familiar all nickel apparatus for preparing (2R,3R,4S)-5-oxo-4,6-bis(perchloryloxy)hexane-1,2,3-triyl triperchlorate.

Theoretical Preparation 65: 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide; Oxidothiiran-2N; 3-oxodiazoxane-2N; Fentyl nitro nitroxy; ENOO



1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide

Expanded structure



3D structure

Molecular data:

Molecular Formula = $C_4H_7N_3O_7S$
Formula Weight = 241.17928
Composition = C(19.92%) H(2.93%) N(17.42%) O(46.44%) S(13.30%)
Molar Refractivity = Not available
Molar Volume = 197 ± 1.690.0 cm^3
Parachor = Not available
Index of Refraction = 1.092 ± 0.0998
Surface Tension = Not available
Density = 1.89 ± 0.099 g/cm^3
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 241.00047 Da
Nominal Mass = 241 Da
Average Mass = 241.1793 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6980	Flammable liquid. Burns with a black smoky flame emitting toxic fumes of sulfur dioxide, nitrogen oxides, and carbon monoxide.	Very stable liquid explosive; non-sensitive to shock, percussion, concussion, heat, pressure, and friction. Requires blasting cap or detonator for proper initiation. A secondary explosive.	Reactivity moderate; is oxidized by strong oxidizers such as potassium permanganate, bleaching powder, and potassium dichromate. Explodes in contact with xenon trioxide—keep away!; does not react with reducing	Colorless, slightly yellow viscous toxic liquid. Boiling point: 180 Celsius; melting point: 2 Celsius. Insoluble in water, alcohol, ethyl acetate, and ether, but slightly soluble in carbon disulfide, 40%

			agents, but the nitro group can be reduced to an amine by reduction with tin and hydrochloric acid.	hydrofluoric acid solution, and liquid ammonia; insoluble in hydrocarbons, toluene, but moderately soluble in methylene chloride or chloroform, soluble in concentrated sulfuric acid.

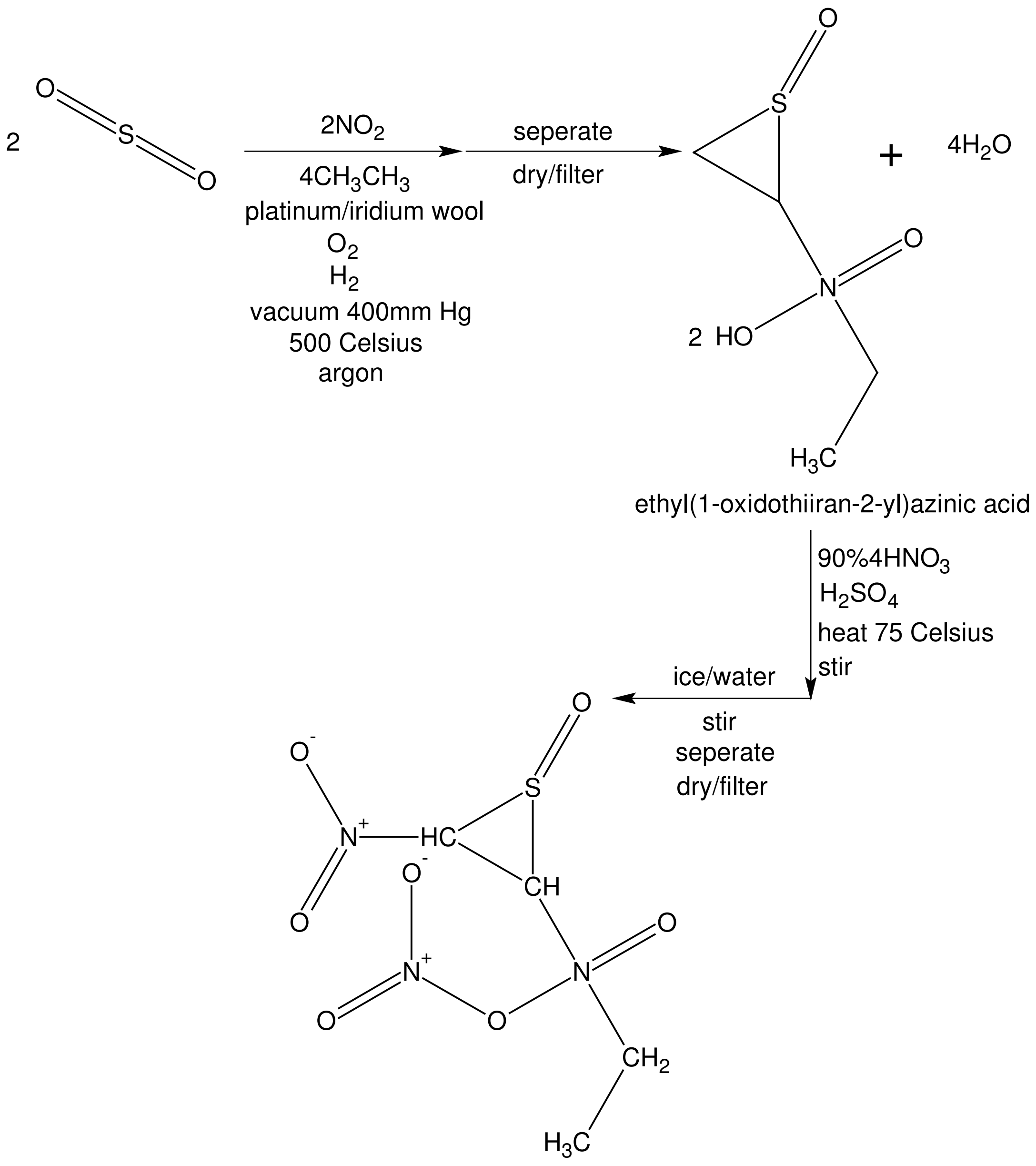
Estimated Compound summary: 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide is a very fascinating and interesting liquid explosive that is extremely stable (20 times then nitroglycerine), and it can be used as a replacement for nitroglycerine and other unstable polyhydric liquid explosives for making dynamites for military and commercial blasting operations when absorbed in powdered aluminum oxide at the ratio of 9 parts 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide, and 2 parts aluminum oxide, and then primed. 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide also forms a jelly like mass that solidifies to a dough-like mass that can be extruded into any desired shape when mixed with nitrocellulose and a high energy plasticizer, and hence can be used to make high performance gun propellants for small arms weapons, propellants for mortar shells, and as a propellant in high caliber munitions such as artillery shells. 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide can be used as an oxidizer in liquid powered rockets, and in hybrid rockets for military use—the explosive has a high gas to solid ration as upon combustion all products of combustion are gasses, and hence the explosive has a high thrust to weight ratio. As with nitroglycerine and other polyhydric liquid explosives 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide should be handled with care and should not be touched by the skin or inhalation of vapors as extreme migraines will result. 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide is rapidly absorbed by the skin forming sores and lesions—use caution when handling. 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide may be a carcinogen so wear gloves and breathing apparatus when handling.

Safety Info: Use maximum ventilation and avoid inhalation of the fumes when handling sulfur dioxide gas, and nitrogen dioxide gas, both of which are toxic and very corrosive. Extinguish all flames before using oxygen gas, ethane gas, and hydrogen gas, as they are highly flammable and can form explosive mixtures when ignited in the presence of the oxygen. Wear gloves and protective mask, and use maximum ventilation when handling 90% red fuming nitric acid, which is highly corrosive, and evolves toxic fumes of nitrogen oxides, avoid skin contact and inhalation of the liquid and fumes. Wear gloves, and wear proper lab clothing when handling concentrated sulfuric acid, which a strong acid and will char many organic substances, avoid skin contact.

Materials:

1. 150 grams of a 60% platinum 40% iridium wool	7. Argon from tank
2. 129 grams of sulfur dioxide gas	8. 250 grams of anhydrous magnesium sulfate
3. 92.5 grams of nitrogen dioxide gas	9. 320 grams of 90% red fuming nitric acid
4. 121 grams of ethane gas	10. 650 grams of concentrated sulfuric acid
5. 63.99 grams of oxygen gas	11. 100 grams of anhydrous sodium carbonate
6. 6.05 grams of hydrogen gas	

Reaction Equation:

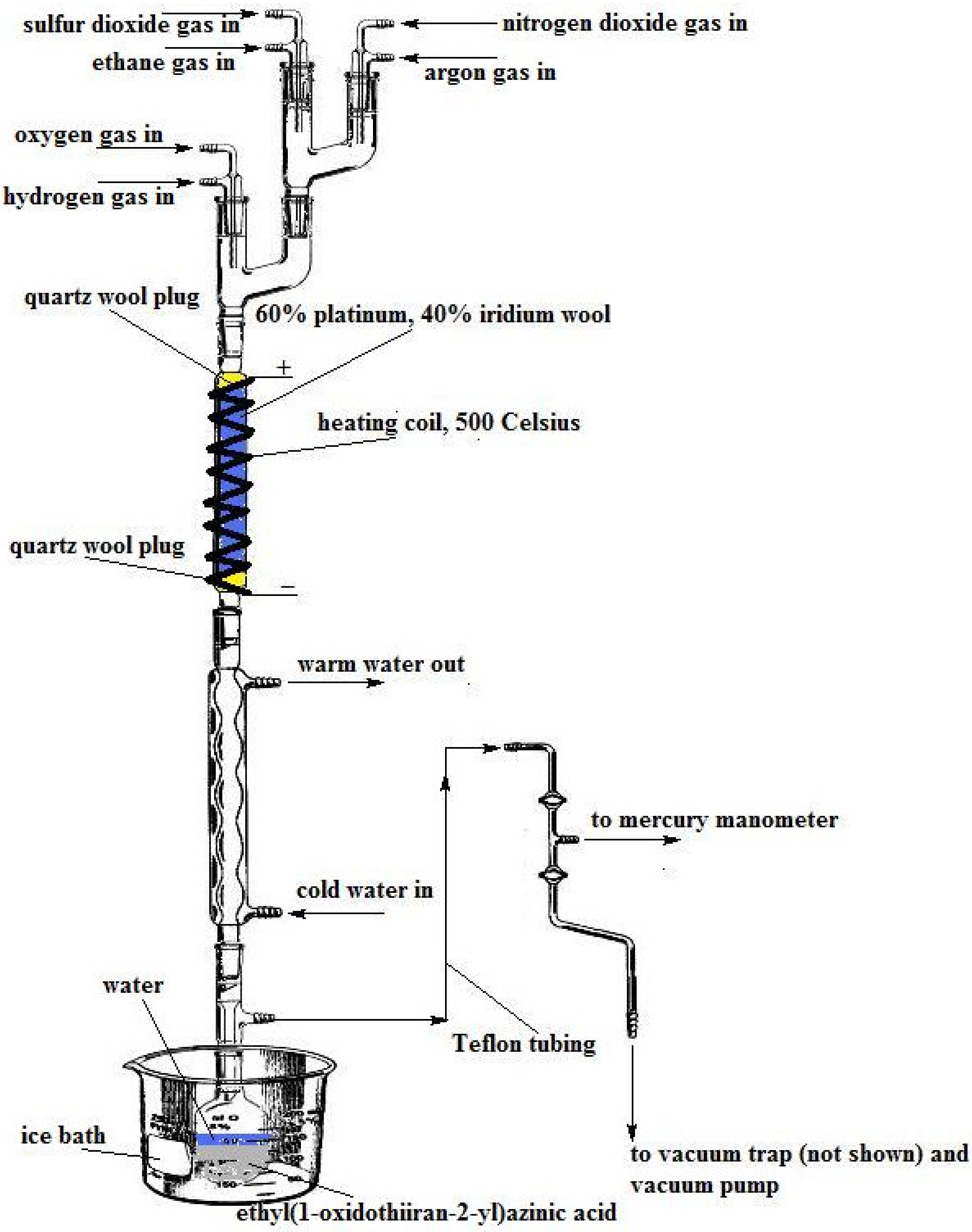


1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide

Preparation:

Step 1: preparation of ethyl(1-oxidothiiran-2-yl)azinic acid

Into an advanced apparatus as illustrated below, place into the catalyst tube 150 grams of a 60% platinum 40% iridium wool and make sure to spread it out to cover the entire length shown in the illustration. Then plug both ends of the catalyst tube with quartz wool as illustrated. Then assemble the heating coil as illustrated, and then begin to pass water through the bulb condenser as shown. Immediately thereafter, turn on the heating coil and while it begins to heat up, begin to pass in argon gas to purge the system. Note: pass in the argon gas at a flow rate of 0.500 mg per/minute. Once the heating coil has reached 500 Celsius, turn on the vacuum pump to 400mm of mercury, and then immediately thereafter begin to pass in a plurality of gases all at the same time by passing in sulfur dioxide gas at a flow rate of 0.09 mg per/minute, nitrogen dioxide gas at a flow rate of 0.064 mg per/minute, ethane gas at a flow rate of 0.084 mg per/minute, followed by oxygen gas at a flow rate of 0.044 mg per/minute, and then finally followed by hydrogen gas at a flow rate of 0.014 mg per/minute. Continue to pass in this plurality of gasses while maintaining a vacuum of 400mm of mercury and maintaining the heating coil at 500 Celsius until 129 grams of sulfur dioxide gas has been added, 92.5 grams of nitrogen dioxide gas has been passed in, 121 grams of ethane gas has been added, 63.99 grams of oxygen gas has been added, and 6.05 grams of hydrogen gas ass been added. Once all the gasses have been added, turn off the heating coil, and allow it to cool to room temperature before removing it, then turn off the vacuum pump and turn off the argon gas purge, and allow the apparatus to come to STP. Once the heating coil has cooled, disassemble the apparatus, and then remove the ice bath, and allow the products in the receiver flask to warm to room temperature. Once it has, pour the reactants in the receiver flask as illustrated into a separatory funnel (not shown), and then drain off and collect the lower desired liquid product of ethyl(1-oxidothiiran-2-yl)azinic acid. Finely, pour this drained-off liquid product of ethyl(1-oxidothiiran-2-yl)azinic acid into a suitable sized beaker, and then add in 100 grams of anhydrous magnesium sulfate (to absorb moisture), and then stir for about 10 minutes. After stirring for ten minutes, filter-off the insoluble magnesium sulfate using a Buchner funnel under strong suction, and then collect the filtered liquid product of ethyl(1-oxidothiiran-2-yl)azinic acid and store in an amber glass bottle in a cool dry place until use for step 2.

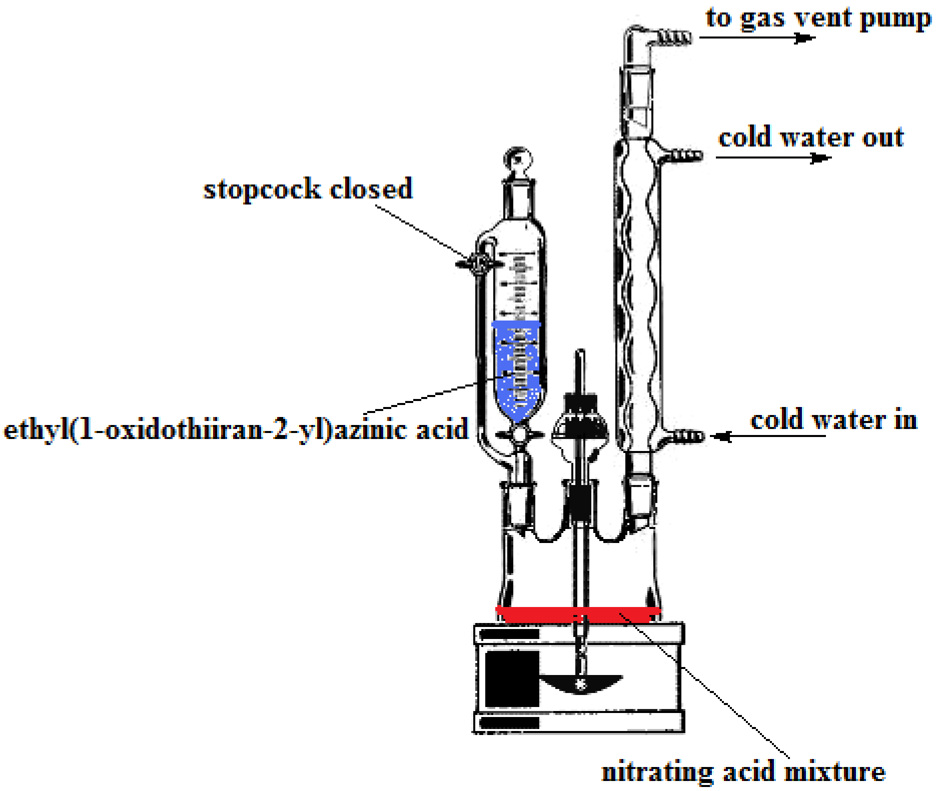


Advanced apparatus for the preparation of ethyl(1-oxidothiiran-2-yl)azinic acid.

Step 2: preparation of 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide

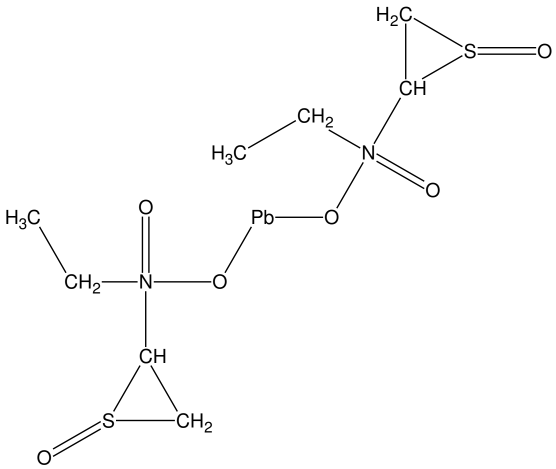
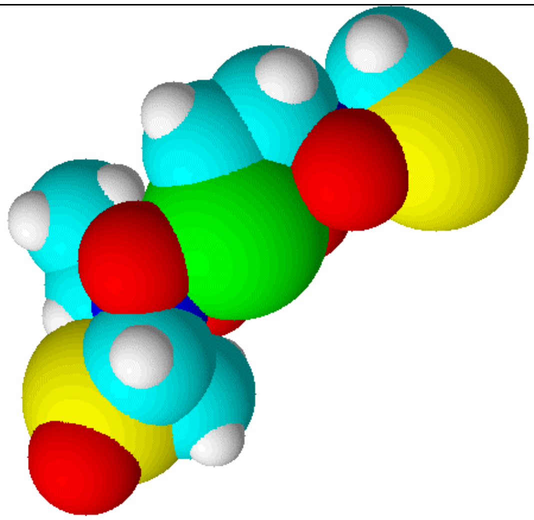
Into an apparatus as illustrated below, pour in 320 grams of 90% red fuming nitric acid into the reaction flask, and then slowly and carefully pour in 650 grams of concentrated sulfuric acid over a period of 1 hour, whiles rapidly stirring the acid mixture. Note: as the sulfuric acid is added red fumes of nitrogen oxides will form, so use maximum ventilation and avoid inhalation of these oxides. Once all the sulfuric acid has been added to the nitric acid, the nitrating acid mixture is ready. Now, turn on the heating mantle to about 75 Celsius under rapid stirring, and when the temperature of the reaction mixture reaches 75 Celsius under rapid stirring, begin to drip in, slowly, the ethyl(1-oxidothiiran-2-yl)azinic acid prepared in step 1 over a period of about

2 hours while rapidly stirring the reaction mixture and maintaining its temperature below 80 Celsius. Note: during the addition of the ethyl(1-oxidothiiran-2-yl)azinic acid to the nitrating acid reaction mixture, the heat will increase, so monitor the temperature of the acid reaction mixture carefully—stopping the addition of the ethyl(1-oxidothiiran-2-yl)azinic acid while at the same time, turning off the heating mantle, may be needed off and on during the entire addition and reaction of the ethyl(1-oxidothiiran-2-yl)azinic acid to keep the temperature of the reaction mixture below 80 Celsius. Now, once all the ethyl(1-oxidothiiran-2-yl)azinic acid has been slowly added to the reaction mixture. Turn off the heating mantle, and allow the reaction mixture to cool to room temperature. Note: during this cool down period, continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, carefully, and very slowly, pour the entire reaction mixture into a large beaker filled with ice. Once the ice has melted, add in more ice, and then stir this entire diluted reaction mixture for about 1 hour, adding more ice as needed. Note: as the ice melts and water forms, a two-phase mixture will result, with the desired product of 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide forming the upper layer, and the acid layer forming the bottom layer. Now, after stirring and adding ice for 1 hour, pour this entire two-phase mixture into a large suitable sized separatory funnel or break the two-phase reaction mixture into fractions (if a large suitable sized separatory is unavailable), and then drain-off the bottom acid layer carefully until all the acid layer has been removed. Once the lower acid layer has been removed, and the upper desired liquid product of 1-ethyl-1-(3-nitro-1-oxidothiiran-2-yl)-3-oxodiazoxane 1,3-dioxide is left over, and collected, pour the collected liquid product into a suitable sized beaker, and then add in 100 grams of anhydrous sodium carbonate (to destroy traces of acid), and then add in 150 grams of anhydrous sodium sulfate (to absorb moisture), and then stir this mixture for 10 minutes. After stirring for 10 minutes, filter-off the insoluble sodium sulfate and any remaining sodium carbonate or acid salts, using a Buchner funnel under strong suction, and then pour the filtered desired liquid product into an amber glass bottle in a dry place until use.



Simple apparatus for nitrating ethyl(1-oxidothiiran-2-yl)azinic acid.

Theoretical Preparation 66: 3,7-bis(1-oxidothiiran-2-yl)-4,6-dioxa-5-plumbous-3,7-diazanonane 3,7-dioxide; bis-OPDPD; azinic acid lead-II-salt; Lead Azinate

 3,7-bis(1-oxidothiiran-2-yl)-4,6-dioxa-5-plumbous-3,7-diazanonane 3,7-dioxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = $C_8H_{16}N_2O_6PbS_2$
Formula Weight = 507.55244
Composition = C(18.93%) H(3.18%) N(5.52%) O(18.91%) Pb(40.82%) S(12.64%)
Molar Refractivity = Not available
Molar Volume = 344.9 ± 09.0 cm^3
Parachor = 709.009 ± 0914.0 cm^3
Index of Refraction = 7.0021 ± 0.97
Surface Tension = 129.07 ± 1.998.0 dyne/cm
Density = 9.005 ± 0.12 g/cm^3
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 554.90635 Da
Nominal Mass = 889 Da
Average Mass = Not available

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:
6731	Non-flammable; explodes when heated to above 100 Celsius.	Sensitive to heat, shock, percussion, concussion, pressure, and friction. Sensitivity almost identical to lead azide. A primary explosive.	Stable towards most chemicals attack, except reacts violently with fluorine, but stable to all the other halogens. Does not react with strong oxidizers or strong bases. Reacts with aqua regia, and concentrated sulfuric	White needle-like crystals; Melting point: explodes when heated; insoluble in water and all known solvents.

			acid with decomposition.	

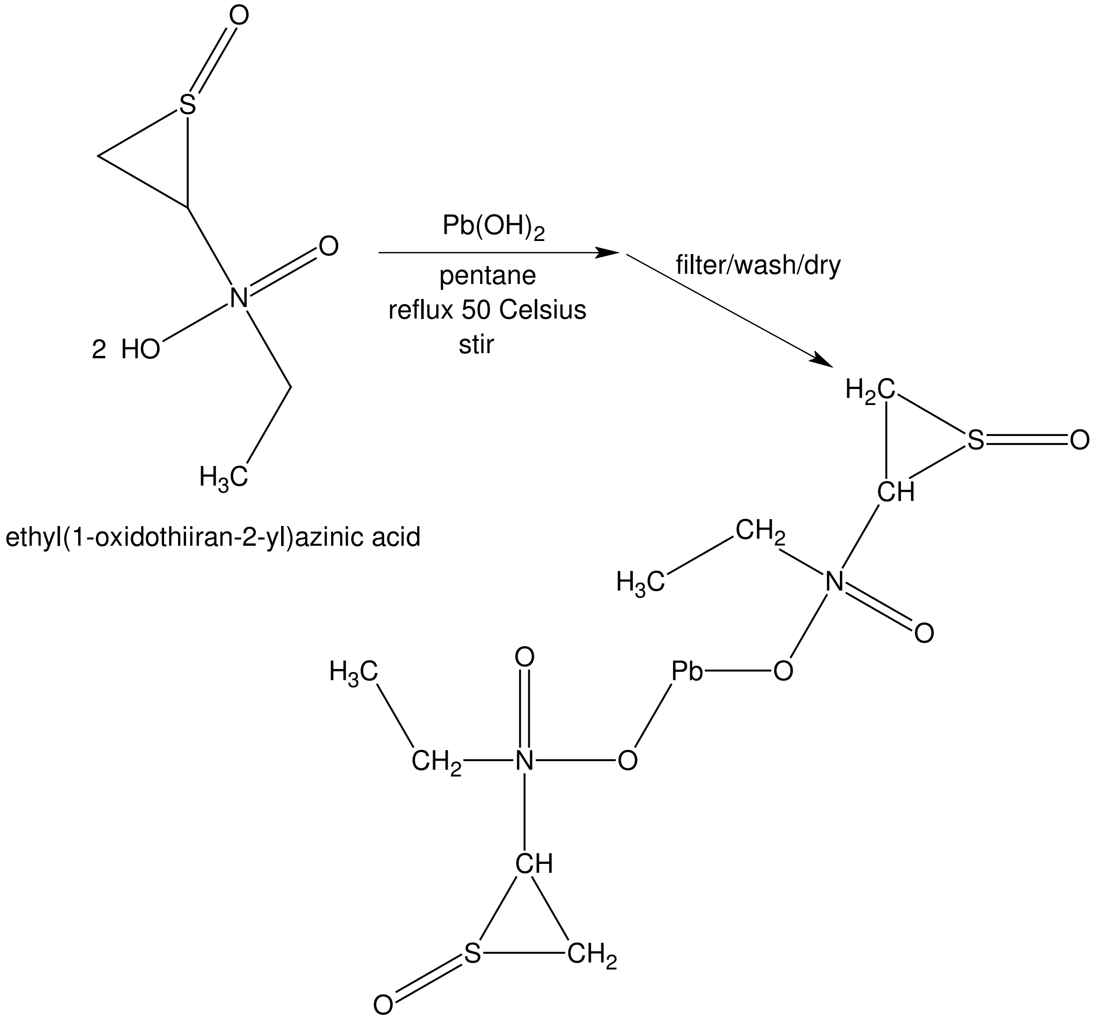
Estimated Compound summary: bis(1-oxidothiiran-2-yl)-4,6-dioxa-5-plumbous-3,7-diazanonane 3,7-dioxide is an interesting lead based azanine explosive with properties almost identical to lead azide. The explosive can be used in place of lead azide when diluted with dextrose for use in blasting caps and detonators, but due to its high cost of production its use in place of lead azide is highly unlikely. bis(1-oxidothiiran-2-yl)-4,6-dioxa-5-plumbous-3,7-diazanonane 3,7-dioxide can be used in specialty munitions as a priming agent when diluted with dextrose at a ratio of 12 parts explosive to 3 parts dextrose.

Safety Info: Extinguish all flames before using pentane, which is highly flammable and volatile—use caution. Use care, and wear gloves and face mask when handling ethyl(1-oxidothiiran-2-yl)azinic acid, which can produce severe migraines if vapors are inhaled, or absorbed through the skin. Ethyl(1-oxidothiiran-2-yl)azinic acid is a possible carcinogen so avoid inhalation and skin contact of the liquid—the liquid also produces sores and lesions if left on the skin.

Materials:

1. 500 milliliters of pentane	3. 80 grams of freshly precipitated lead-II-hydroxide
2. 101 grams of ethyl(1-oxidothiiran-2-yl)azinic acid (prepared in theoretical preparation 65, step 1)	

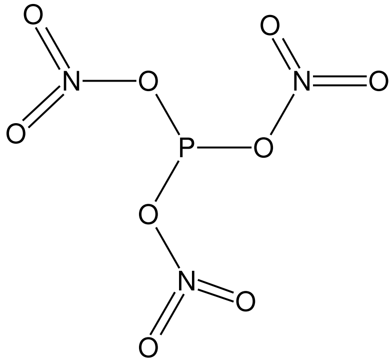
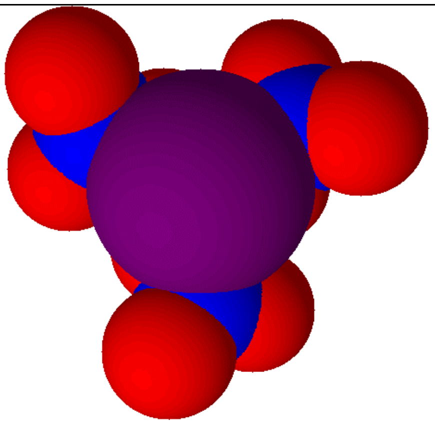
Reaction Equation:



3,7-bis(1-oxidothiiran-2-yl)-4,6-dioxa-5-plumbous-3,7-diazanonane 3,7-dioxide

Preparation: Into a standard reflux apparatus equipped with motorized stirrer, reflux condenser and thermometer, place 500 milliliters of pentane, and then add in all at once 101 grams of ethyl(1-oxidothiiran-2-yl)azinic acid (prepared in theoretical preparation 65, step 1), followed by adding in all at once 80 grams of freshly precipitated lead-II-hydroxide. Thereafter, turn on the heating mantle to approximately 50 Celsius under vigorous stirring, and bring the reaction mixture to a gentle reflux. Then continue to rapidly agitate the reaction mixture under gentle reflux at 50 Celsius for 12 hours. Note: do not exceed a temperature of 70 Celsius. After heating under reflux under vigorous agitation for 12 hours, turn off the heating mantle and allow the reaction mixture to cool to room temperature. Note: during the cool down period, continue to rapidly stir the reaction mixture. Once the reaction mixture has cooled to room temperature, turn off the motorized stirrer, and then disassemble the reflux apparatus, and then filter-off the insoluble solid using a Buchner funnel under gentle suction, and then wash with three 500-milliliter portions of warm water, and then suction dry as best as possible. Finnaly, place the washed filtered-off product onto a shallow pan and allow it to thoroughly air-dry for 7 days. Once the desired product is dry, collect the crystals and place them into a plastic container and store in a cool dry place until use.

Theoretical Preparation 67: [phosphanetriyltris(oxy)]tris(oxoazane) trioxide; Phosphorus trinitrate; PTN-170; Nitroxy phosphorus

 [phosphanetriyltris(oxy)]tris(oxoazane) trioxide	
Expanded structure	3D structure

Molecular data:

Molecular Formula = N_3O_9P
Formula Weight = 216.988462
Composition = N(19.37%) O(66.36%) P(14.27%)
Molar Refractivity = Not available
Molar Volume = Not available
Parachor = Not available
Index of Refraction = Not available
Surface Tension = Not available
Density = Not available
Dielectric Constant = Not available
Polarizability = Not available
Monoisotopic Mass = 216.937215 Da
Nominal Mass = 217 Da
Average Mass = 216.9885 Da

Molecular Information:

Detonation velocity (estimated by monoisotopic mass and index of refraction):	Estimated Flammability:	Estimated Sensitivity:	Reactivity:	Estimated Physical aspect:

7009	Non-Flammable liquid. Does not form ignitable mixtures with reducing agents.	Remarkably very stable. Non-sensitive to shock, percussion, concussion, heat, pressure, or friction. Requires blasting cap or detonator for proper initiation. A secondary explosive.	Generally very resistant to chemical attack. However, can be oxidized by strong oxidizers when heated. Has no reaction with strong bases, or acids.	Colorless transparent water-white viscous liquid. Boiling point: 178 Celsius; melting point: 3 Celsius. Insoluble in water, alcohol, and ethyl acetate, but soluble in ether, methylene chloride, but only slightly soluble in hydrocarbon solvents. Note: this compound is a known cholinesterase inhibitor, with effects like, yet less then parathion—avoid skin contact, ingestion, and inhalation.

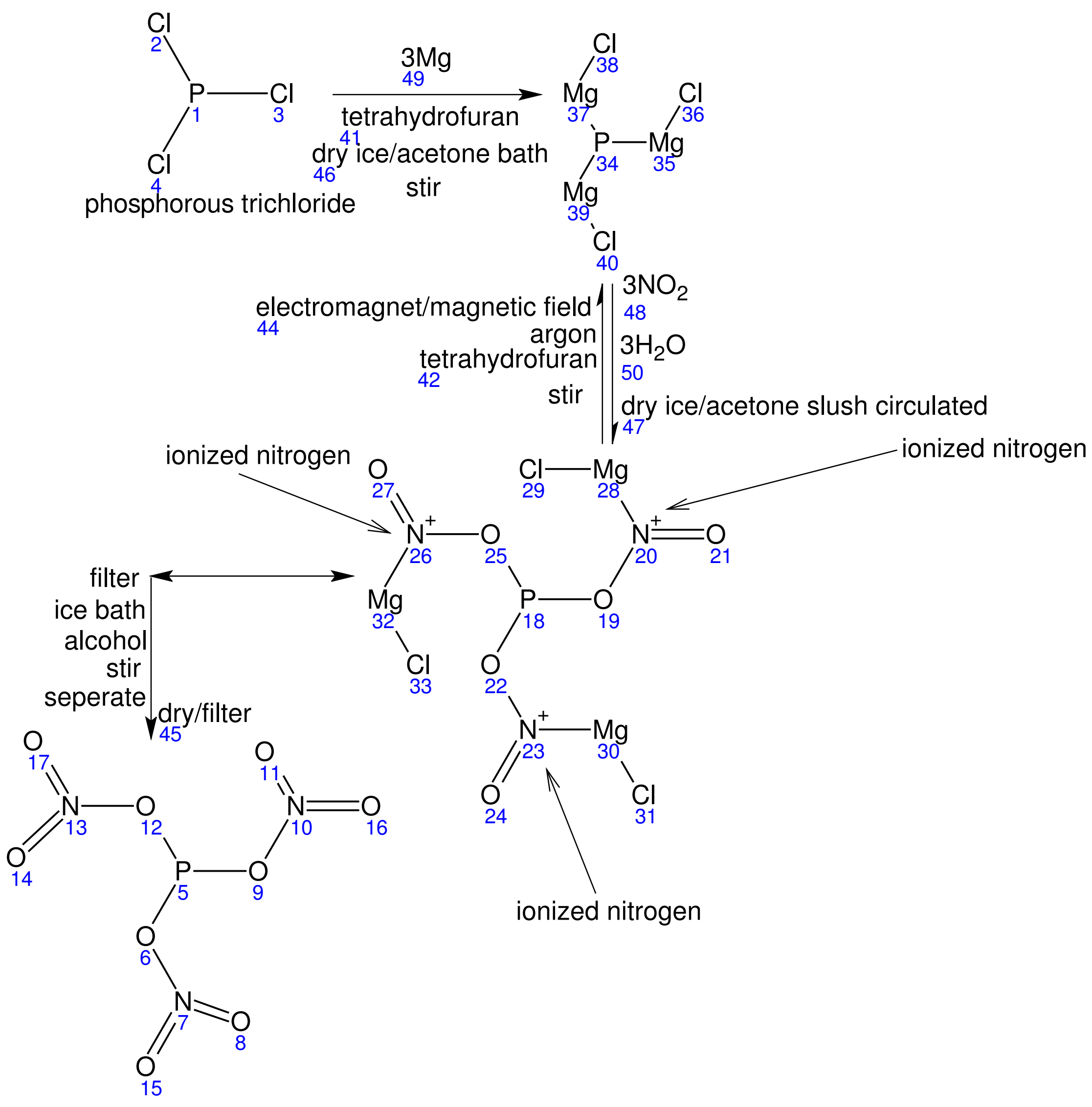
Estimated Compound summary: [phosphanetriyltris(oxy)]tris(oxoazane) trioxide is an interesting phosphorus-to-oxygen nitroxy nitrate explosive liquid. Its properties are amazing, and it has remarkable stability for a liquid explosive, i.e., the polyhydric nitrate explosive like nitroglycerine. [phosphanetriyltris(oxy)]tris(oxoazane) trioxide can be used directly by itself and poured directly as a liquid into mortar shells, artillery shells, missile warheads, canister warheads for rockets, and many other uses; however, in cold weather conditions the liquid explosive will freeze so hence a small amount of propylene glycol should be added to the liquid explosive to lower its melting point. Unfortunately, [phosphanetriyltris(oxy)]tris(oxoazane) trioxide does not get absorbed by any known substance such as diatomaceous earth, aluminum oxide, ect., but it can form a jelly like mass when mixed with a small amount of aluminum palmitate, gasoline, and nitrocellulose. This jelly-like product can then be used in filling paper or cardboard tubes for making blasting dynamites for military and commercial blasting operations and for mining operations involving either soft rock, or hard rock blasting operations. [phosphanetriyltris(oxy)]tris(oxoazane) trioxide does not poses the proper physical properties to be used as an oxidizer in liquid fueled rockets, and it does not react with reducing agents even when heated therefore it cannot be used to make rocket fuels or gun propellants, and pyrotechnic mixtures.

Safety Info: Use proper ventilation and avoid inhalation of the vapors when handling tetrahydrofuran, which is highly volatile and flammable—tetrahydrofuran is a narcotic in high concentrations, avoid skin contact and inhalation. Note: although tetrahydrofuran is a cyclic ether, it is capable of forming dangerous peroxides, so never use tetrahydrofuran that has been in storage for prolonged periods of time without performing the peroxide test. Wear gloves when handling phosphorus trichloride and avoid skin contact an inhalation of the fumes. Phosphorus trichloride reacts violently with water and alcohols. Use maximum ventilation when handling nitrogen dioxide, which is very toxic; avoid inhalation of the fumes at all cost.

Materials:

1. 750 milliliters of tetrahydrofuran	5. 132 grams of dry nitrogen dioxide gas
2. 131 grams of phosphorus trichloride	6. Argon gas from tank
3. 70 grams of clean magnesium turnings (can be obtained by quickly soaking magnesium turnings into dilute hydrochloric acid, followed by washing with water, and thoroughly drying).	7. 750 milliliters of 99% isopropyl alcohol
4. 57 milliliters of distilled water	8. 75 grams of anhydrous sodium sulfate

Reaction Equation:

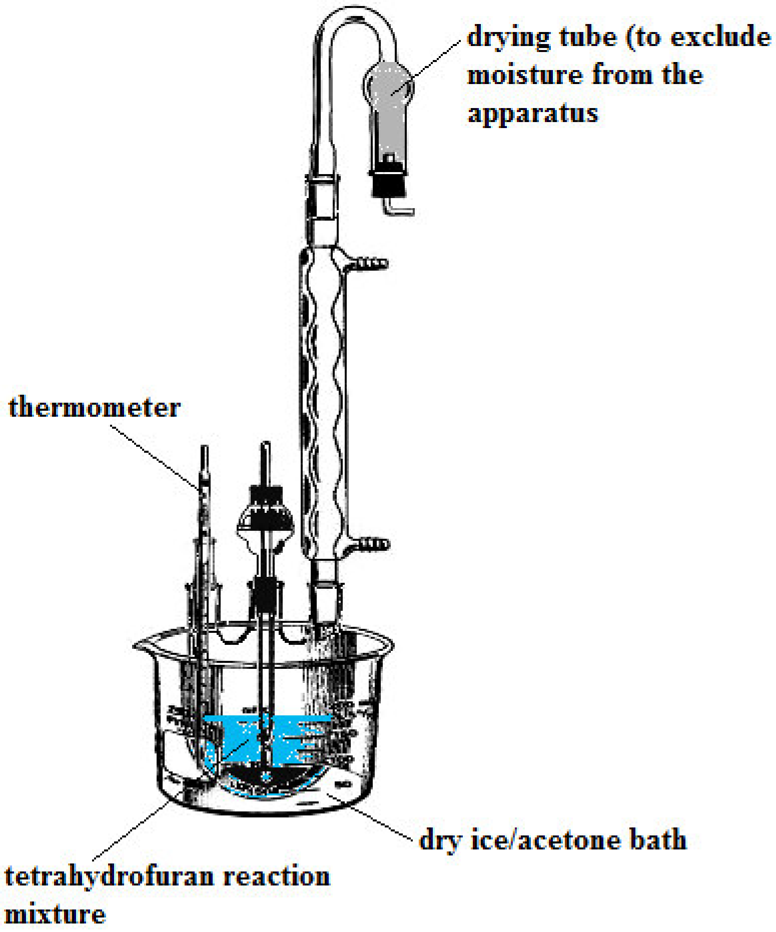


[phosphanetriyltris(oxy)]tris(oxoazane) trioxide

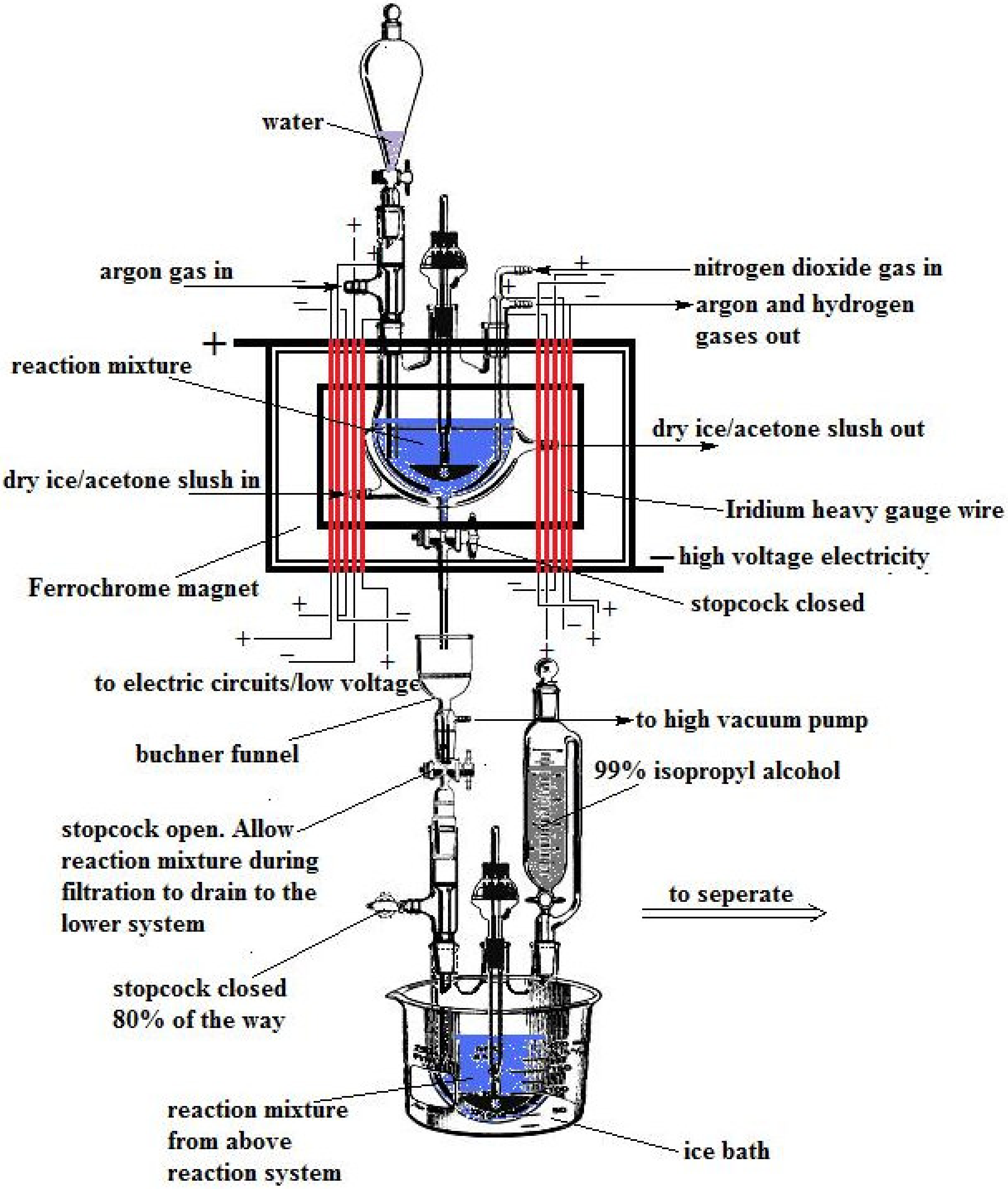
Preparation:

Step 1: preparation of the Grignard Reagent and the desired product [phosphanetriyltris(oxy)]tris(oxoazane) trioxide

Into a Grignard reaction apparatus as illustrated below, place 750 milliliters of tetrahydrofuran, followed by adding in 131 grams of phosphorus trichloride, followed by adding in 70 grams of clean magnesium turnings, and then seal the apparatus, and then turn on the motorized stirrer and stir the reaction mixture on rapid speed for approximately 4 hours. During this 4-hour reaction time period, a Grignard reagent will form (a Grignard reagent is R-MgX). After 4 hours, the Grignard reagent would have formed, so remove the dry ice/acetone bath, and allow the reaction mixture to warm to room temperature. Once the reaction mixture has warmed to room temperature, quickly disassemble the apparatus and pour the entire reaction mixture into the advanced apparatus illustrated below (the second apparatus illustrated). Now, pour 57 milliliters of distilled water into the upper separatory funnel as illustrated in the second advanced apparatus. Thereafter, attach the black negative clamp to the negative pole of the ferrochrome magnet, and then attach the red positive clamp to the positive pole of the ferrochrome magnet. Make sure the iridium heavy gauge wire is in place as illustrated and attached to a circuit control board (not illustrated), and this circuit control board attached to 10 nine volt batteries and turn on the circuit board and allow the electricity to flow through the iridium heavy gauge wire. Thereafter, turn on the electricity to the ferrochrome magnet using a voltage of 10,000 volts at 1 amp DC, and then immediately begin to pump a acetone dry ice slush through the outer cooling jacket as illustrated, and then begin a argon gas purge at a flow rate of 0.750 mg per/minute, and then wait 5 minutes to flush out the air, and then begin to pass in dry nitrogen dioxide gas at a flow rate of 0.250 mg per/minute, and continue to cool and turn on the motorized stirrer to high speed, and continue to pass in dry nitrogen dioxide gas until 132 grams of dry nitrogen dioxide gas has been passed into the reaction mixture. Once all the desired amount of dry nitrogen dioxide gas has been added, continue the operation with the desired voltage systems, while continuing to cool and stir the reaction mixture for 1 hour. Now, after 1 hour, turn off and pump out the dry ice acetone slush cooling agent, and then open the stopcock to the upper separatory funnel containing the distilled water as illustrated, and allow a slow drip of the distilled water into the reaction mixture while continuing a argon purge and while continuing to rapidly stir the reaction mixture. Note: continue the electricity to the ferrochrome magnet and the circuits at the desired voltage during the addition of the distilled water. Once all the distilled water has been added to the reaction mixture, continue to stir the reaction and continue the electricity to the ferrochrome magnet and circuits at the desired voltage for 1 hour. After 1 hour, turn off all electricity, and then turn off the motorized stirrer, and then open the lower stopcock of the reaction 3-neck flask, and allow the reaction mixture to slowly drain into the Buchner funnel. Immediately thereafter, turn on the vacuum pump to maximum during the filtration process. Note: the insoluble by-product of magnesium hydroxy chloride will collect in the Buchner funnel during the draining and suction filtration process. Continue to slowly drain the upper reaction mixture into the Buchner funnel under suction, until all of the original reaction mixture has passed through the Buchner funnel and drained into the lower 3-neck reaction flask contained in an ice bath, and equipped with another motorized stirrer and addition funnel. Then pour in 750 milliliters of 99% isopropyl alcohol into the addition funnel as illustrated, and then turn on the second motorized stirrer, and then begin a moderate drip of the alcohol into the reaction mixture that is now contained in the lower 3-neck flask reaction system as illustrated. Note: once the alcohol begins to be added to the reaction mixture the desired liquid product will begin to separate out forming a lower layer. Once all the alcohol has been added, continue to rapidly stir the reaction mixture for 1 hour with or without the ice bath. After 1 hour, disassemble the apparatus, and then pour the two-phase reaction mixture into a large separatory funnel, or if a large separatory funnel in unavailable, divide the two-phase reaction mixture into smaller portions and use a proper sized separatory funnel for each fraction. Now, slowly drain-off the bottom liquid layer, and then place this drained-off bottom liquid layer into a suitable sized beaker, or if you had to divide the two-phase reaction mixture into smaller portions, collect each drained-off lower layer into the same suitable sized beaker. Finnaly, to the liquid desired product contained in the suitable sized beaker, add in 75 grams of anhydrous sodium sulfate (to absorb moisture), and then stir this mixture for 45 minutes. After 45 minutes, filter-off the sodium sulfate using a Buchner funnel under suction, and then collect the filtered liquid desired product of [phosphanetriyltris(oxy)]tris(oxoazane) trioxide and store it in an amber glass bottle in a cool dry place until use.


drying tube (to exclude moisture from the apparatus
thermometer
dry ice/acetone bath
tetrahydrofuran reaction mixture

Classic setup for preparing a Grignard Reagent.


water
argon gas in
nitrogen dioxide gas in
argon and hydrogen gases out
reaction mixture
dry ice/acetone slush out
dry ice/acetone slush in
Iridium heavy gauge wire
high voltage electricity
Ferrochrome magnet
stopcock closed
to electric circuits/low voltage
to high vacuum pump
buchner funnel
99% isopropyl alcohol
stopcock open. Allow reaction mixture during filtration to drain to the lower system
to seperate
stopcock closed 80% of the way
reaction mixture from above reaction system
ice bath
+
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Super advanced apparatus for preparing [phosphanetriyltris(oxy)]tris(oxoazane) trioxide.

References:

Stand alone book. No references cited to protect potential or possible future copyrighted material, pending patents, and corporate research and development operations and procedures. This book is experimental and does not reflect any known or slightly, moderately, or thoroughly researched explosives listed in this book, and hence, references are non-existent at this point in time, circa 2010.